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ABSTRACT

This technical specialty course is one of thirty-five courses designed for use by two-year postsecondary institutions in five nuclear technician curriculum areas: (1) radiation protection technician, (2) nuclear instrumentation and control technician, (3) nuclear materials processing technician, (4) nuclear quality-assurance/quality-control technician, and (5) nuclear reactor (plant) operator trainee. This course, Advanced Radionuclide Analysis, contains seven modules: Survey of Methods Used in Radionuclide Analysis (Sample Collection and Analysis), Gamma Spectrometry, Alpha Spectrometry, Beta Spectrometry, Radiochemical Methods of Analysis by Liquid Scintillation Counting, Neutron Activation Analysis, and Determination of Specific Nuclides (in the Environment). Each module follows a typical format including the following sections: introduction, module prerequisites, objectives, subject matter, exercises, materials, procedures, data tables, reference materials, and tests. (A master index and suggested program planning guides are also available--see CE 026 856 and CE 026 639-643.) (LRA)

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ED192104

Nuclear Technology Series

COURSE 22

ADVANCED RADIONUCLIDE ANALYSIS

CENTER FOR OCCUPATIONAL RESEARCH

AND DEVELOPMENT, INC.

4800 LAKEWOOD DRIVE

WACO, TEXAS 76710

CE 026 662

U S DEPARTMENT OF HEALTH
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PREFACE

ABOUT NUCLEAR TECHNOLOGY MODULES

The modules – developed for use in two-year postsecondary technical institutions to prepare technicians for employment – are useful in industry for updating employees in company-sponsored training, and as laboratory texts in engineering schools. The principles, techniques and skills taught in the modules, based on tasks that nuclear technicians perform, were obtained from a nationwide advisory committee of employers of nuclear technicians. Each module was written by a working technical expert/experienced writer team and tested with students.

A module contains the following elements:

Introduction, which identifies the topic and often includes a rationale for studying the material.
Module Prerequisites, which identify the material a student should be familiar with before studying the module.
Objectives, which clearly identify what the student is expected to know for satisfactory module completion. The objectives, stated in terms of action-oriented behaviors, include such action words as operate, measure, calculate, identify and define, rather than words with many interpretations, such as know, understand, learn and appreciate.
Subject Matter, which presents the background theory and techniques supportive to the objectives of the module. Subject matter is written with the technical student in mind.
Exercises, which provide practical problems to which the student can apply this new knowledge.
Materials, which identify the equipment required to complete the laboratory procedure.
Procedures, which is the experiment section, or "hands-on" portion of the module (including step-by-step instruction) designed to reinforce student learning.
Data Tables, which are included in most modules for the first year (or basic) courses to help the student learn how to collect and organize data.
Reference Materials, which are included as suggestions for supplementary reading/viewing for the student.
Test, which measures the student's achievement of the prestated objectives.

A helpful Index has been added at the end of each course.

The materials contained herein were developed pursuant to a contract with the Office of Education, U.S. Department of Health, Education and Welfare: Contract No. 300780582. Contractors undertaking such projects under Government sponsorship are encouraged to express freely their professional judgment in the conduct of the project. Points of view or opinions stated do not, therefore, necessarily represent official Office of Education position or policy.

The Nuclear Technology Series consists of five curricula:

Nuclear Reactor (Plant) Operator Trainee.
Radiation Protection Technician.
Nuclear Materials Processing Technician.
Nuclear Quality-Assurance/Quality-Control Technician.
Nuclear Instrumentation and Control Technician.

The completed Nuclear Technology Series consists of the following 35 technical specialty courses:

Radiation Physics.
Radiation Protection I.
Principles of Process Instrumentation.
Heat Transfer and Fluid Flow.
Introduction to Nuclear Systems.
Instrumentation and Control of Reactors and Plant Systems.
Reactor Operations.
Reactor Safety.
Reactor Auxiliary Systems.
Power Plant Systems.
Radiation Detection and Measurement.
Reactor Physics.
Power Plant Chemistry.
Introduction to Quality Assurance/ Quality Control.
Metallurgy and Metals Properties.
Mechanical Component Characteristics and Specifications.
Radiation Protection II.
Radiological Emergencies.
Radiation Shielding.
Radiation Monitoring Techniques (Radiochemical).
Radioactive Materials Disposal and Management.
Advanced Radionuclide Analysis.
Nuclear Chemical Processes.
Nuclear Systems and Safety.
Radioactive Material Handling Techniques.
Nondestructive Examination (NDE) Techniques I.
Metrology.
Welding Inspection.
Civil/Structural Inspection.
Mechanical Inspection.
Quality-Assurance Practices.
Nondestructive Examination (NDE) Techniques II.
Control Systems I.
Control Systems II.
Power Plant Electrical Systems and Components.

Availability: Instructional modules are available individually or by courses from:

Center for Occupational Research and Development
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Waco, Texas 76710
Telephone (817) 772-8756

Printed:

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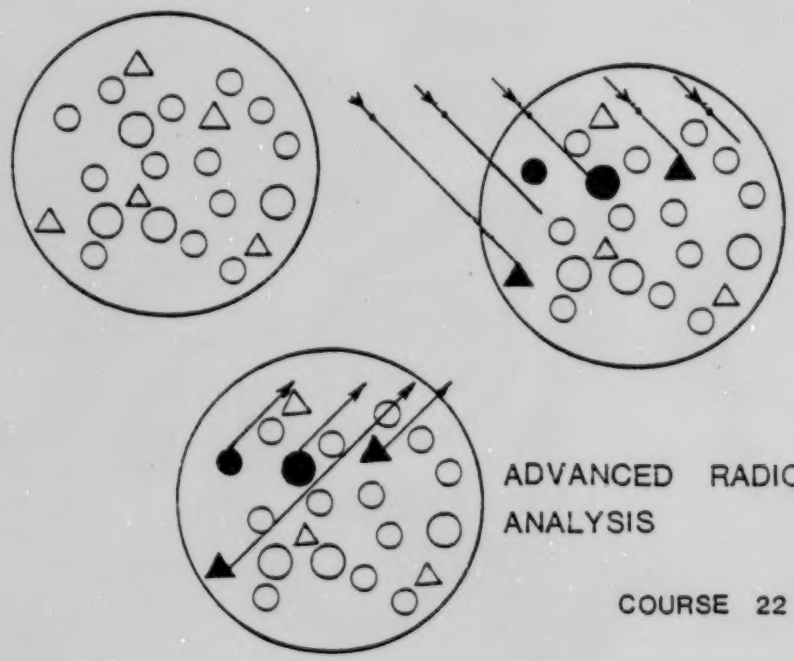
NUCLEAR TECHNOLOGY

MODULE 22-1

SURVEY METHODS USED IN RADIONUCLIDE ANALYSIS
(SAMPLE COLLECTION AND ANALYSIS)



ADVANCED RADIONUCLIDE ANALYSIS

COURSE 22

ORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 22 ADVANCED RADIONUCLIDE ANALYSIS

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INTRODUCTION

The sampling of environmental releases from a nuclear facility is a sound procedure and is required by local, state, and federal agencies. Radionuclide samples are collected for analysis in order that the following questions can be answered:

- What nuclides are present?
- In what materials are the nuclides present? (This material is called the matrix.)
- What quantity of each relevant nuclide is present?

This module, the first in this series, details methods of collection for environmental sampling. The other modules in this course specify methods of analysis.

MODULE PREREQUISITES

The student should have completed Course 1, "Radiation Physics."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Describe three types of environmental releases.
2. Define the following terms:
   Continuous sample.

Noncontinuous or grab sampling.
Matrix.
Filtration sampling.
Condensation.
Electrostatic precipitation.
Impaction sampling.
Absorption.
Ashing.
Dry-weight activity.
Ion-exchange process.
Eluant.
Ion-exchange resin.

3. Identify and describe four methods of collecting radionuclides found in air; and identify any listed specific uses, advantages, and disadvantages.
4. Describe the chemical method used to separate or to fixate a gas sample in the field.
5. List the precautions that must be taken when air and water samples are taken from around a facility.
6. Describe the procedures that must be followed when obtaining samples of dirt or sediment.
7. State the methods used to concentrate biological material.
8. State the advantages of expressing activities of foods as dry-weight activity.
9. Describe the ion-exchange process.
10. Describe procedures for preventing absorption of radionuclides in liquid samples.
11. List data that should be included in a sampling notebook.

ENVIRONMENTAL RADIATION SURVEILLANCE

Environmental radiation surveillance is conducted for a number of important reasons, including the following ones:

- To provide information for the assessment of dose exposure of a population.
- To test the adequacy of source control measures.
- To follow radiation tracers in research studies.
- To observe gross trends in air or water.
- To measure radionuclide concentrations in specific media.

There are several different types of radioactive contamination that should be considered separately. Radioactive materials may be transported from one location to another by means of movement of air masses. The air may be contaminated either with radioactive gases or particles of solid or liquid radioactive materials, usually in the form of invisible dust. Water and food taken into the body by mouth may be contaminated with radioactive materials.

Radioactive material is transferred from the atmosphere by meteorological processes, particularly by rain. The most serious contamination problems arise from direct deposition of the radionuclides upon animal feed crops or upon food crops directly consumed by man. Following the initial deposition upon vegetation, the radioactive materials tend to be removed by various processes: they may be washed off by rain or be blown off by wind. The extent to which such removal occurs depends upon a number of considerations, including particle size, chemical properties of the material deposited, and environmental or biological factors.

For any particular radionuclide, the critical pathway, or route, by which it reaches man must be known. Then samples can be collected for it, and estimates of its effects upon man can be made.

SAMPLING PROGRAMS OR SURVEYS

Many factors must be considered before a sampling program is put into effect. The kind of program required depends upon the specific objectives, ranging from routine surveillance for evaluating general radiation levels to special programs for specific radionuclides important in assessing radiation exposure of populations.

Many of the problems involved in surveillance are related to the size and variability of the natural environment. The transmission pathways of radioactive material through the terrestrial food chain to man are illustrated in Figure 22-1-1.

SAMPLING OBJECTIVES

Depending on the objectives of the particular sampling program, measurement of the concentration of radionuclides in the air breathed by persons in a city, county, or state may be necessary. The necessity may arise to evaluate the activity concentration of radionuclides in milk in a milkshed containing thousands of cows and serving many thousands of people. Sampling may be required to determine the activity concentration in a large, rapidly-flowing river or a lake or an estuary, or to evaluate the radionuclide intake of a population (and the resulting dose) by analyzing air, water, or food samples.

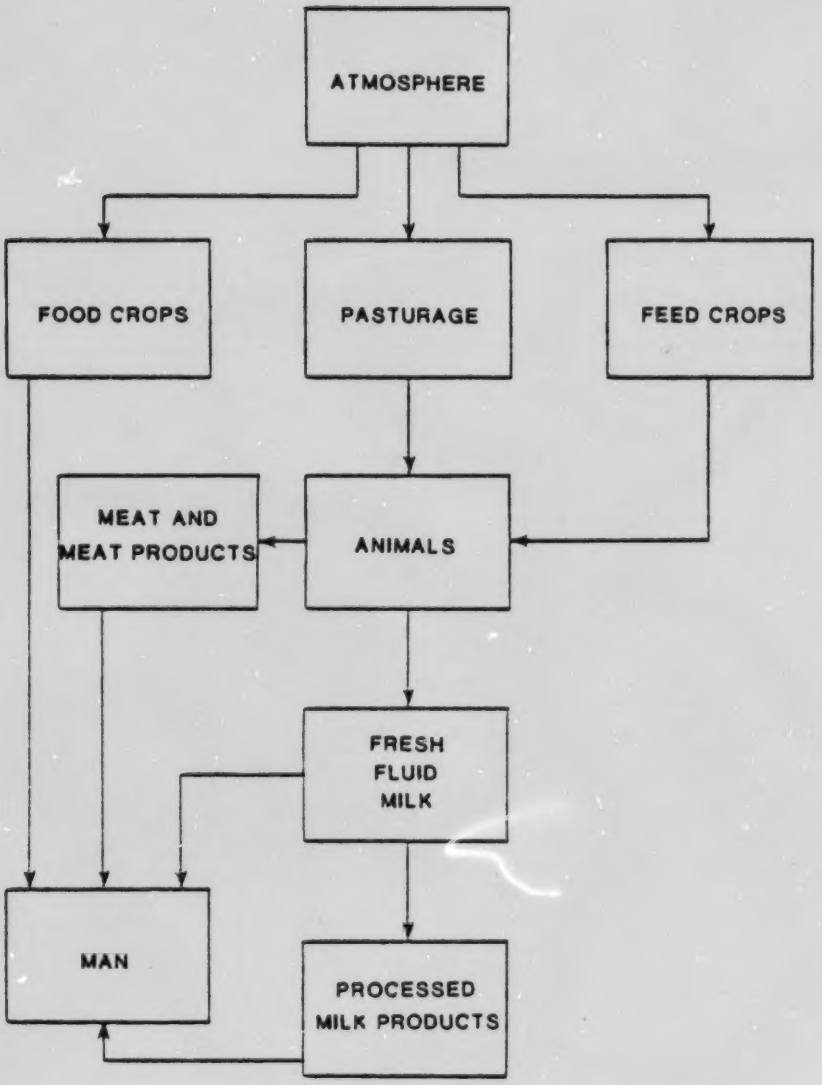

Figure 22-1-1. Important steps in the transmission of radioactive material through the food chain to man.

In establishing the objectives of a sampling program, the following items, among others, must be considered: variety in terrain, meteorology, agricultural practices, diet, and economic situations from one area to another in a state or country.

Unless the survey is planned and executed properly, the result may be a meaningless accumulation of data (or too much or too little data), rather than an orderly, logical program that produces useful information.

If there is a high possibility for the release of a specific nuclide, a media that will concentrate this nuclide should be sampled. One nuclide may be more critical because of local dietary habits and levels of discharge. If this is the case, it may be necessary to sample only a limited number of media.

SOURCES AND DISPERSION OF RADIONUCLIDES

Possibly eighty to ninety percent of all fallout materials reaching the earth is brought down in precipitation. The waters in streams, however, contain only about one percent of the activity present in the precipitation. The major contributors to the aquatic environment, therefore, are precipitation directly deposited on streams or lakes, and liquid wastes from reactor operations, chemical processing, and radionuclide users.

Radionuclides in water may gain access to the diet through aquatic plants and animals, either by surface contamination or by biological uptake. The latter is influenced by the concentration of radionuclides, the stable mineral content of the aquatic environment, and the position of a particular plant or animal in the aquatic food chain. Vegetative organisms, such as phytoplankton and filamentous algae, which absorb nutrients directly from the water, accumulate the greatest concentration of radioactive material. Next are the zooplankton and bottom animals which feed on the plant minerals, and then the juvenile and small fish

that feed principally on these animals. At the end of the chain are the game and food fish that, in turn, feed on the juveniles.

The sources of radioactive contaminants in the environment include natural radioactivity, uranium ore mining and milling, reactor operations (including chemical processing of spent fuels), facilities that use radioisotopes, and weapons tests. Samples must be taken in areas of these operations to ensure safety.

THE FUNDAMENTAL PROBLEM OF SAMPLING

The fundamental problem in sampling is to determine to what extent the sample is representative of the material being sampled. The selection of the sampling point itself may influence the result of the measurement.

SAMPLING PROCEDURES

There are two types of environmental monitoring: continuous and noncontinuous. The environment can be monitored continuously by measuring devices that constantly measure a physical parameter and record the magnitude for permanent records. No chemical separations are done, and this method usually cannot approach the sensitivity of a rigorous chemical analysis.

Noncontinuous sampling is the preferred method of detecting minute traces of radionuclides in the environment. In this method, a sample of gas, liquid, or solid material is obtained and the material analyzed by the methods described in this module and in the modules to follow. This

method sometimes is known as "grab sampling." The sample obtained must be representative of typical radionuclide concentrations at the sampling point. Numerous techniques are used to insure that this is so. Precautions must be taken to protect the sample from contamination; and, because radionuclide concentrations in the environment are usually very low, chemical processes must be utilized to concentrate the sample prior to analysis.

There are three types of analysis; they correspond to the three states of matter. Special procedures and equipment are employed to obtain samples of gases, liquids, and solid material.

The first step in sampling is the establishment of a sampling strategy. The technician must decide either to sample for a specific radionuclide or to sample for any or all radionuclides. When sampling for a specific radionuclide, the technician obviously looks in the places in which it might be found in natural systems. A typical soil-sample problem is illustrated in the example that follows.

EXAMPLE	
Given:	A soil sample in which some radionuclides are present.
Find:	(a) The total amount of beta-radioactivity present or (b) the total amount of ^{131}I activity present.
Solution:	(a) The sample is dried, sieved (sifted), and prepared for counting. It then is counted by use of a beta-sensitive counter. If the count rate, the counting efficiency, and the weight of the sample are known, the specific

activity of the sample can be determined. No determination of specific radionuclides is made, for the beta activity may be caused by any one of, or combination of, the following beta-emitting radionuclides: ^{90}Sr, ^{40}K, ^{226}Ra, ^{3}H, ^{131}I,.... .

(b) The sample is dried and weighed. It then is dissolved and iodine-chemistry performed. The ^{131}I is counted, and the extent of its disintegration rate is determined. A report prepared for such an analysis would give the amount of ^{131}I present per unit weight of material.

In general, a specific radionuclide is sought where it likely will be found and/or to do the most damage; for example, the rare gases Xe and Kr would be found primarily in the air around a facility. The isotopes of iodine often are sought in the milk from cows grazing near a facility, although the more soluble radionuclides such as Na, Cs, and Rb are searched for in their dissolved state in water.

Specific methods for sampling the three phases of matter are given in the next three sections.

GASEOUS SAMPLES

Gaseous radionuclides and radionuclides suspended in air can be collected by the following methods:

- Filtration.
- Condensation.

- Electrostatic precipitation.
- The use of impactor systems.

The most widely-used method of air sampling is filtration. Air is pumped through a filter made of fiberglass, cellulose asbestos, or plastics. The volume rate and length of time is recorded. At the end of a specified time period, the pump is turned off, and the filter is placed into a sample collection container and labeled for future counting and analysis.

Two types of sampling pumps are in frequent use: (1) A vacuum-cleaner type (Figure 22-1-2) that has a relatively small filter diameter (about 6 cm) and air suction capacity (about 1 m^3/h); and (2) larger centrifugal blowers that have filter diameters of about 20 cm and suction capacities of 20 to 30 m^3/h. Small battery-operated systems often are used.

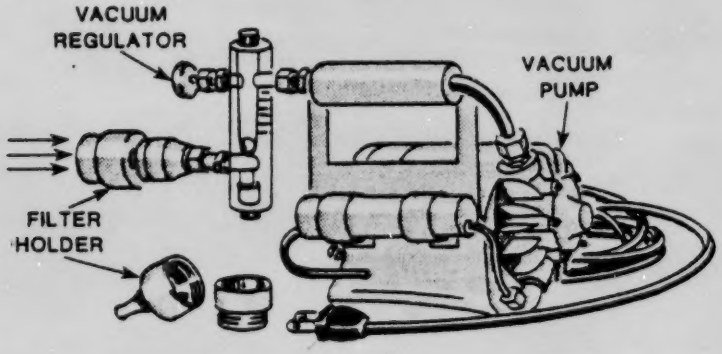

Figure 22-1-2. Typical air-sampling pump.

In continuous collection, a filter strip generally is used. The strip passes through a sampling chamber, either at a constant feed rate or in steps. Figure 22-1-3 illustrates the principle of one design that has been used in practical applications. (Collection by electrodeposition is rarely used in continuous measurement.)

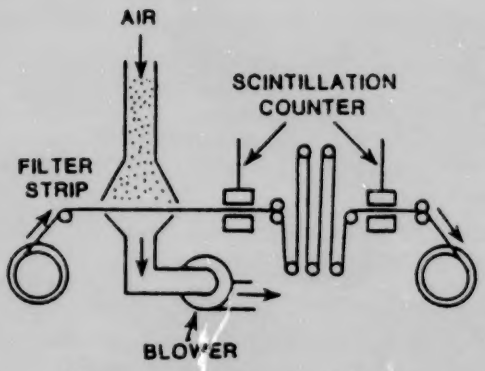

Figure 22-1-3. Typical continuous-measurement air monitor.

After an appropriate delay period, the activity carrier passes one or more detectors, i.e., end-window counters or scintillation counters. When scintillation counters are utilized, a special design of scintillation phosphor that has a slot through which the filter strip passes sometimes is used to increase the measuring sensitivity. In this way approximately 4π-geometry is obtained. The filter strip must be insensitive to moisture in order to avoid expansion or tearing in humid air.

Since it is not usually possible to carry out an integrated measurement of the amount of air that has flowed through a moving filter strip, the throughput of sampled air must be controlled accurately.

Many continuous monitors contain strip-charts in order that radiation level is recorded continuously.

Fission products of inert gases such as xenon or krypton can be removed by cooling the air to a very low temperature to remove the condensable gases. This process is called "condensation."

Electrostatic precipitators can be used to collect ionized particles. In its simplest form, it consists of an electrically-charged wire suspended in the air (Figure 22-1-4). Collection efficiency is low since only particles

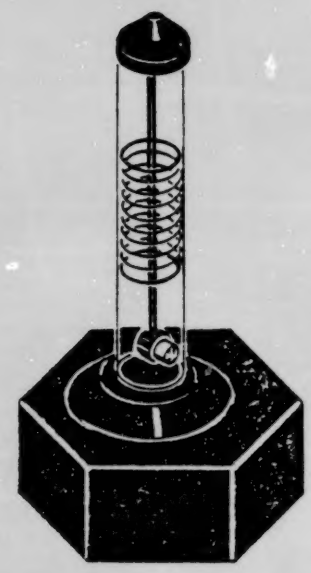

Figure 22-1-4.
Electrostatic precipitator
(demonstration model).

that already are ionized are attracted to the wire. One type of precipitator consists of a mechanism that draws air through a tube in which a centrally-located electric wire has been stretched. When voltage is applied to the wire, radionuclides suspended in aerosol form precipitate on the inner surface of the tube. The tube then can be connected directly to an ionization chamber, in which the tube contents are counted.

Electrostatic precipitators operate upon the principle that most tiny dust particles attract free electrons and thereby acquire a negative electrical charge. As the particles pass a positively-charged plate, they are attracted to it. Some electrostatic precipitators are constructed with the attractive plate in the form of a cylinder lined with a thin metallic foil, and with the negative electrode, a centrally located wire, as an axis. As the air passes through

the cylinder, the particles are attracted to the foil, which then can be removed from the cylinder for measurement.

Air flow can be directed onto a prepared surface to precipitate heavy dust particles by inertia. Oil and vaseline often are used as the adhesive agent. This method of collection is called "impaction sampling."

When gases are collected in the field, chemical separation of fixation must be done immediately in the field. One method often used is adsorption – the process whereby a solid, such as charcoal, condenses and holds a gas upon its surface. The gas may or may not be chemically separated from the adsorbing material when it is analyzed for radionuclides.

When taking air samples from around a facility, the technician must be careful to sample the air both up and downwind of the facility. The upside can act as the control; the downside will represent the control plus the contaminants the facility has contributed. The technician must be certain that the upwind sample is not contaminated by particles borne upwind by local eddy currents.

SOLID SAMPLES

One of the greatest problems in the collection of solid samples is uncertainty concerning how much of the matrix to collect. Generally, a relatively large sample of matrix should be collected because the concentration of almost all radionuclides is rather low, measured in the normal concentration units of activity of radionuclide per volume or mass of matrix.

The most common solids collected are dirt and sediment. These, as a general rule, should be taken from the surface. All vegetative matter should be removed and the sample strained through a sieve before being dried. The sample then is dried out in a drying cabinet or placed in a glass beaker and carefully heated until they completely dry. After the material is dried, it can be counted and analyzed.

Biological material (plants and animals) is a natural collector of activity that provides information on the distribution of nuclides over an area or over a specific period time. Concentration of biological material is achieved by drying and ashing (the reduction of the material to ashes). Because some radioactive isotopes may become vaporized during this process, biological substances often are concentrated by dissolving them in acid. This concentration process usually reduces the volume, but sometimes increases it.

When the sample is analyzed, the activity can be indicated in pCi/g of fresh or dry weight. Since moisture contents of foods differ, the activity of a foodstuff should be cited in pCi/g of dry weight when comparisons are made. Dry-weight activity also is valuable since tables for natural radioactivity per gram of dry substance are available. This activity can be subtracted as background radioactivity that naturally occurs when the sample is analyzed. Natural activity in biological samples is almost exclusively that of the potassium isotope ^{40}K. The ^{40}K content of plants is about 20 pCi/g of dry substances.

LIQUID SAMPLES

The majority of radioactive waste is liquid. Most decontamination procedures, cleaning processes, and chemical

activities produce radioactively-contaminated liquids, usually water. Radioactivity can be transported in water over large distances without there being any great dilution of the activity. In this way, contaminated industrial or drinking water can exist even some distance from the original point of contamination and potentially can endanger its users. For these reasons, the sampling of water is especially important and acquires additional significance when there is a possibility that radioactive material may be introduced into bodies of water from which drinking supplies are taken. Experience has shown that conventional filters used to process drinking water possess low decontamination factors for many radioactive nuclides.

When sampling water, technicians must ensure that the samples are representative ones. Both upstream and downstream samples should be taken, and the selection of the sampling point must be made carefully; for example, when the water in a river is sampled, technicians must be certain to sample the running water as well as the stagnant water of pools and channels. A number of samples should be taken of stagnant water since activity is not usually distributed uniformly. Before wastewater is sampled, the contents of the wastewater tank should be mixed.

Several methods are used to prepare samples from liquids. If the radionuclide is in the form of insoluble particles, it is filtered out. If the radionuclide is in the form of a gas, the liquid is degassed; and the removed gases are passed through a drying agent to remove all moisture. If the radionuclide is in a solution, the dissolved ions in the liquid are removed by an ion-exchange process.

Ion-exchange processes remove ions in liquids that contain small numbers of ions. Such liquids include water

in reactor cooling systems and water that contains relatively minute amounts of radionuclides per unit volume, as does river water downstream from a nuclear facility. In this method, ion-exchange resin bonds to specific ions. In a typical procedure, an ion-exchange resin column is constructed (Figure 22-1-5), and the liquid is allowed to pass through the ion-exchange resin. The column either can be counted directly, or the ions can be removed by the passage of a specially-prepared solution called the "eluant."

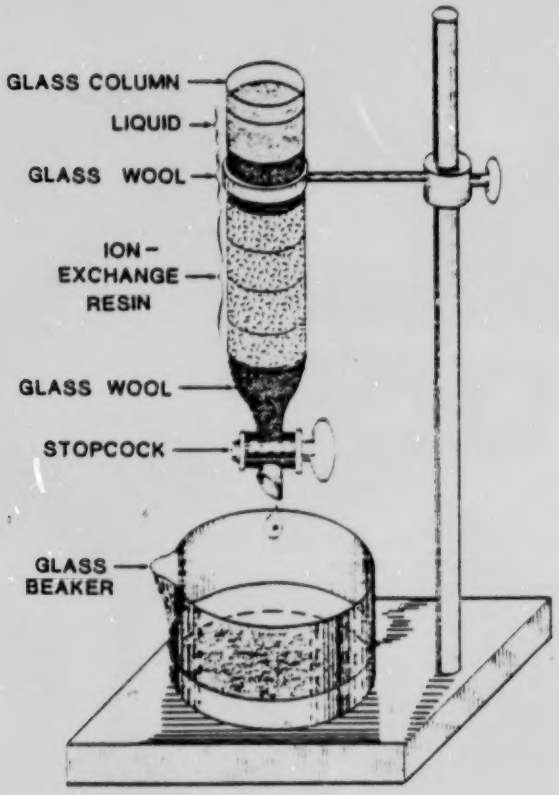

Figure 22-1-5.
Ion-exchange resin column.

The ion-exchange resin is an insoluble electrolyte that usually has one heavy ion and an oppositely-charged simple ion that can be exchanged for ions in the liquid. Both natural and synthetic materials are used as ion-exchange resins.

One difficulty involved with liquid samples is the tendency of the ions to become adsorbed on the glass walls of containers and on new and unused plastic vessels. Adsorption to metal vessels and rubber tubes also is high. Cesium and strontium-90 are two of the many radionuclides that tend to deposit on surfaces. This deposition can be prevented if the solution is made acidic by the addition of 1% by volume HNO_3 or $HC\ell$. Plastic containers should be used whenever possible.

The natural activity in water may disturb measurements, especially when activities that are lower than the natural activity of water must be measured. Most of the natural activity in water is made up of the activities of radon and thorium and their daughters. These radionuclides diffuse into the water from the uranium and thorium minerals in the soil. Potassium ^{40}K occasionally is found in water samples.

Ion-exchange processes are extremely dependent upon the quality of the water sampled. Since only a certain quantity of ions can be bound to a given amount of ion-exchange resin, the method is applicable mainly to water containing an absolutely small number of ions, i.e., essentially to precipitated water and, in nuclear engineering, to the control of reactor cooling systems. When handling ion exchangers, care should be taken that the exchanger substance behaves like a strongly acid or alkaline substance. The disadvantage of this method is that the activity is bound to a relatively large mass of ion-exchange material;

therefore, for β-measurement one must either elute or take into account the high self-absorption of the ion-exchanger mass. Another drawback is the long time required for the procedure, since the flow rates through ion exchangers must be kept small to avoid channelling. On the other hand, separation of radionuclides is possible; and this method facilitates subsequent identification of the individual emitters.

RECORD KEEPING

All releases from nuclear facilities are monitored by regulatory branches of government, including local and state departments of Health and Environmental quality, and federal agencies such as the NRC, EPA, OSHA, and DOE. Data Tables 22-1-1 through 22-1-5 are typical of the type of records that must be kept. Typical examples from the regulations covering waste disposal are given in Figure 22-1-6 and the accompanying Table 22-1-1. Because each facility must be able to document the extent of its releases, accurate record keeping is extremely important. The technician conducting an environmental sample must keep a notebook, which should include the following data:

- Date and the time of the sample.
- Location.
- Reason for sample (routine or special).
- Full names and titles of all persons performing and observing the procedure.
- A description of all sampling techniques used.

WASTE DISPOSAL

Sec.C.301 General Requirement. No licensee shall dispose of any radioactive material except:

(a) By transfer to an authorized recipient as provided in Part B, or

(b) As authorized pursuant to Sections C.106 or C.303.

(c) As authorized under the provisions of Oregon Revised Statutes.

Sec.C.303 Disposal by Release Into Sanitary Sewerage Systems. No licensee shall discharge radioactive material into a sanitary sewerage system unless:

(a) It is readily soluble or dispersible in water; and,

(b) The quantity of any radioactive material released into the system by the licensee in any one day does not exceed the larger of subparagraphs (1) or (2) of this paragraph:

(1) The quantity which, if diluted by the average daily quantity of sewage released into the sewer by the licensee, will result in an average concentration not greater than the limits specified in Appendix A*, Table I, Column 2, of this Part; or

(2) Ten times the quantity of such material specified in Appendix B of this Part; and

(c) The quantity of any radioactive material released in any one month, if diluted by the average monthly quantity of water released by the licensee, will not result in an average concentration exceeding the limits specified in Appendix A*, Table I, Column 2, of this Part; and,

(d) The gross quantity of radioactive material released into the sewerage system by the licensee does not exceed one Curie per year.

Excreta from individuals undergoing medical diagnosis or therapy with radioactive material shall be exempt from any limitations contained in this Section.

*A portion of Appendix A is contained in Table 22-1-1.

Figure 22-1-6. Typical extract from regulations on waste disposal.

TABLE 22-1-1.

TYPICAL DISPOSAL TABLES TO BE USED WITH REGULATIONS IN FIGURE 22-1-6.

(A portion of Appendix A referenced in Figure 22-1-6.)

Element (atomic number)	Isotope[1]		Disposal Column 1 Air (μCi/ml)	Disposal Column 2 Water (μCi/ml)
Lanthanum (57)	La-140	S	2×10^{-7}	7×10^{-4}
		I	1×10^{-7}	7×10^{-4}
Lead (82)	Pb-203	S	3×10^{-6}	1×10^{-2}
		I	2×10^{-6}	1×10^{-2}
	Pb-210	S	1×10^{-10}	4×10^{-6}
		I	2×10^{-10}	5×10^{-3}
	Pb-212	S	2×10^{-8}	6×10^{-4}
		I	2×10^{-8}	5×10^{-4}
Lutetium (71)	Lu-177	S	6×10^{-7}	3×10^{-3}
		I	5×10^{-7}	3×10^{-3}
Manganese (25)	Mn-52	S	2×10^{-7}	1×10^{-3}
		I	1×10^{-7}	9×10^{-4}
	Mn-54	S	4×10^{-7}	4×10^{-3}
		I	4×10^{-8}	3×10^{-3}
	Mn-56	S	8×10^{-7}	4×10^{-3}
		I	5×10^{-7}	3×10^{-3}
Mercury (80)	Hg-197m	S	7×10^{-7}	6×10^{-3}
		I	8×10^{-7}	5×10^{-3}
	Hg-197	S	1×10^{-6}	9×10^{-3}
		I	3×10^{-6}	1×10^{-2}
	Hg-203	S	7×10^{-7}	5×10^{-4}
		I	1×10^{-7}	3×10^{-3}
Molybdenum (42)	Mo-99	S	7×10^{-7}	5×10^{-3}
		I	2×10^{-7}	1×10^{-3}
Neodymium (60)	Nd-144	S	8×10^{-11}	2×10^{-3}
		I	3×10^{-10}	2×10^{-3}
	Nd-147	S	4×10^{-7}	2×10^{-3}
		I	2×10^{-7}	2×10^{-3}
	Nd-149	S	2×10^{-6}	8×10^{-3}
		I	1×10^{-6}	8×10^{-3}
Neptunium (93)	Np-237	S	4×10^{-12}	9×10^{-5}
		I	1×10^{-10}	9×10^{-4}
	Np-239	S	8×10^{-7}	4×10^{-3}
		I	7×10^{-7}	4×10^{-3}
Nickel (28)	Ni-59	S	5×10^{-7}	6×10^{-3}
		I	8×10^{-8}	6×10^{-4}
	Ni-63	S	6×10^{-8}	8×10^{-4}
		I	3×10^{-7}	2×10^{-3}

EXERCISES

1. List and explain four of the reasons why environmental radiation surveillance is necessary.
2. Describe the methods for taking gaseous, solid, and liquid samples.
3. What is the fundamental problem in sampling?

MATERIALS

Natural collection areas such as –

Field.

Stream, lake, or ocean.

Clean vessels for collection of sediment, soil, or liquids.

Clean digging tools (small trowel or spatula).

Balance: capable of up to 2 Kg with a sensitivity of 1 g.

Graduate cylinder: 2 liters.

Set of sieves, sizes 10–30.

Oven – 110°C.

Reagent-grade concentrated HCl or HNO_3.

Ion-exchange resin, Amberlite IRN-150 or Dowex 50W-X8, 20–50 mesh.

Glass ion-exchange column.

Glass wool.

Air filter setup.

Air pump.

THE ION-EXCHANGE PROCESS

The purpose of these procedures is to provide the student with experience in the collection of environmental samples to be submitted to radionuclide analysis.

1. Use Data Tables 22-1-1 and 22-1-2. Collect from the sampling field or the sediment layer of a stream, lake, or ocean a representative sample of dirt. Remove the vegetative matter, and sieve the sample to obtain a homogenous collection. Place sample in a glass beaker and heat at 110°C until completely dry. Place sample in a properly-marked holding container or prepare for counting following the instructions of the instructor. Complete Data Tables 22-1-1 and 22-1-2.
2. Use Data Tables 22-1-3 and 22-1-4. Collect about 2 liters of natural waters. Filter through a rather coarse filter paper (for example Whatman No. 1). Make acidic by adding 1% by volume HNO_3 or HCl. Divide sample in half; place one-half into a properly-labeled plastic storage bottle.
3. Complete Data Table 22-1-3 and applicable portion of 22-1-4.
4. Use Data Table 22-1-4. The other half is to be treated with ion-exchange resin. Construct an ion-exchange resin column such as that illustrated in Figure 22-1-7; use glass wool, a resin selected by the instructor, and a glass column. Be sure that the resin does not have the opportunity to run dry during construction.
5. Lower the excess head of water from the top of the beads, and load the sample carefully so as not to disturb the resin bed any more than necessary.

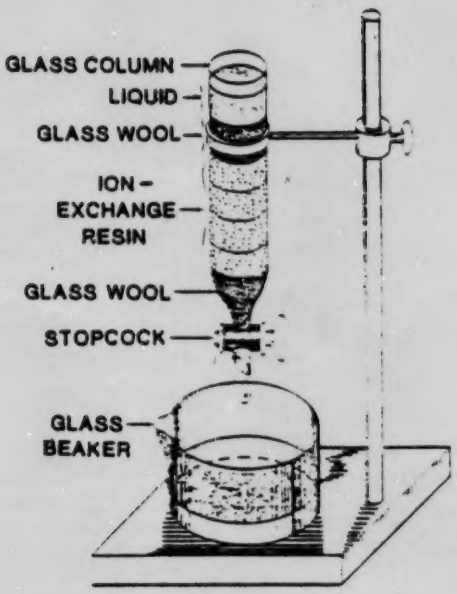

Figure 22-1-7. Ion-exchange resin column.

6. Place the ions of the liquid onto the resin by allowing the liquid to drip through the column at a very slow rate. The rate of liquid coming off the column should be controlled to about 2 ml per min.
7. At this point, the column either can be counted directly or the material thereon eluted off by changing the pH of the eluting solution.
8. Complete Data Table 22-1-4.

AIR SAMPLING BY FILTRATION

1. Use Data Table 22-1-5. Arrange the air pump and air filter apparatus as illustrated in Figure 22-1-8.

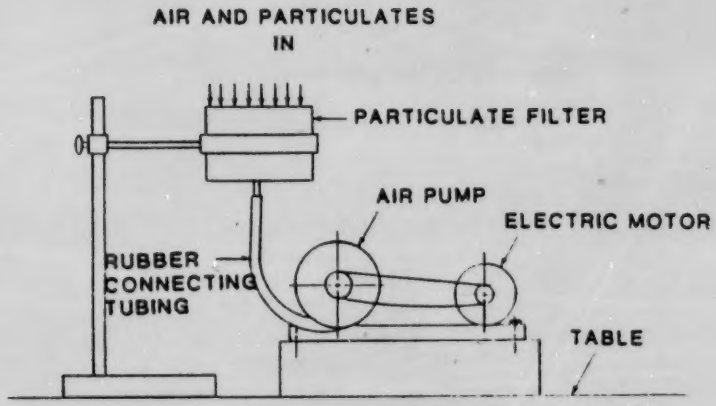

Figure 22-1-8. Arrangement of air sampler system.

2. Turn on pump and allow it to draw air through the filter for at least one hour.
3. Record volume rate, length of time, and liter per min, respectively.
4. At the end of the time period, turn off the pump and carefully place the filter into a sample collection container for future counting. Label container.
5. Complete Data Table 22-1-5.

DATA TABLE 22-1-1
FIELD PROCEDURES FOR FIELD SAMPLE

Date _____	Persons performing sample
Time _____	_____
Location of sample _____	_____
_____	_____
Depth of soil from which	Persons observing samples
sample removed _____	_____
_____	_____
	_____
Equipment used _____	Type of container _____
_____	_____

Field procedures used (such as sieving of filtering).

Approximate mass of sample

DATA TABLE 22-1-2
LABORATORY PROCEDURES FOR SOIL SAMPLE

Mass before drying _____

Dry mass _____

DATA TABLE 22-1-3

FIELD PROCEDURES FOR LIQUID SAMPLE

Date _____	Persons performing sample
Time _____	_____
Location of sample _____	_____
_____	_____
Equipment used _____	Persons observing samples
_____	_____
Type of container _____	_____
_____	_____

Field procedures used (such as filtering).

Approximate volume of sample _____

DATA TABLE 22-1-4

LABORATORY PROCEDURES FOR LIQUID SAMPLE

Volume of sample _____

Type filter used _____

% volume of HNO_3 or HCl Added _____

Volume to be treated with
ion-exchange resin _____

Rate of liquid from column _____

DATA TABLE 22-1-5

FIELD PROCEDURES FOR AIR SAMPLE

Persons performing sample	Persons observing sample
_____	_____
_____	_____
_____	_____
Location of sample _____	Type air pump & air filter
_____	apparatus _____
Volume rate _____	_____
(liters per minute)	Time equipment on _____
Type of container _____	_____
_____	_____

REFERENCE MATERIALS

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Blatz, Hanson. Introduction to Radiological Health. New York: McGraw-Hill Book Company, 1964.

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Mullins, J. W. "The Problem of Radiation Surveillance." Operational Aspects of Radiation Surveillance. Washington, D.C.: U.S. Department of Health, Education, and Welfare, Public Health Service, 1967.

Wang, C. H.; Willis, D. L.; and Loveland, W. D. Radio-tracer Methodology in Biological, Environmental and Physical Sciences. Englewood Cliffs, NJ: Prentice-Hall, 1975. Chapter 18.

Section I: Multiple Choice

Circle the correct answer or answers.

1. The three types of environmental releases are –
   a. Aerosol, wastewater, and gaseous.
   b. Solid, liquid, and gaseous.
   c. Gaseous, liquid, and precipitate.
   d. Geochemical, trace element, and organic.
2. The four methods of collecting radionuclides found in air are –
   a. Adsorption, absorption, precipitation, and concentration.
   b. Filtration, adsorption, condensation, and precipitation.
   c. Condensation, adsorption, impaction, and filtration.
   d. Impaction, precipitation, condensation, and filtration.
3. The method used to fixate a gas in the field is to –
   a. Pass it through an ion-exchange resin.
   b. Use an adsorbent material.
   c. Cooling to remove the condensable gases.
   d. Electrostatic precipitation.
4. Which of the following precautions should be followed when air and water samples are collected in a routine survey?
   a. Wear radiation-protective clothing, and handle all samples with remote-handling devices.
   b. Take samples only downstream and downwind of the facility.

c. Take samples downstream and downwind as well as upstream and upwind.

d. Take local eddy currents into account.

5. Which of the following procedures should one follow when obtaining samples of dirt or sediment?

   a. Take as small a representative sample as possible.

   b. Take sample from as close to the surface as possible.

   c. Be certain to strain sample before drying it.

   d. Use clean vessels as containers.

6. Which of the following methods are used to concentrate biological material?

   a. Ashing.

   b. Drying.

   c. Condensation.

   d. Precipitation.

7. Why should activities in food be stated as a function of dry weight?

   a. Makes comparisons of activity of different foodstuffs easier.

   b. The drying of food concentrates activities.

   c. Natural radioactivity of water interferes with counting.

   d. Dry-weight activities remain constant. Fresh-weight activities vary with freshness of food.

Section II: Matching

Match the terms in Column A with the definitions in Column B by placing the correct alphabetical notation in Column A.

Column A	Column B
_____ Grab sample.	A. Used to remove ions from ion-exchange resin.
_____ Filtration sampling.	B. Useful in removing fission products of inert gases.
_____ Electrostatic precipitation.	C. Materials that contain radionuclides.
_____ Adsorption.	D. Precipitates heavy dust particles by inertia.
_____ Dry-weight activity.	E. Method of concentrating biological material.
_____ Ion-exchange process.	F. Insoluble electrolyte.
_____ Eluant.	G. Constant measuring of a physical parameter.
_____ Ion-exchange resin.	H. Used in preparing samples of liquids with low nuclide concentration.
_____ Ashing.	I. Valuable in comparison of activity of foodstuffs.
_____ Impaction sampling.	J. Method of "fixing" gas.
_____ Matrix.	K. Used to collect ionized particles present in gases.
_____ Condensation.	L. Means of isotope dilution.
_____ Continuous sample.	M. Most widely-used method of gaseous sampling.
	N. Preferred method of detecting minute quantities of radionuclides.

Section III: Complete the following statements:

1. The ion-exchange process is used to separate radionuclides by – _____
_____
_____

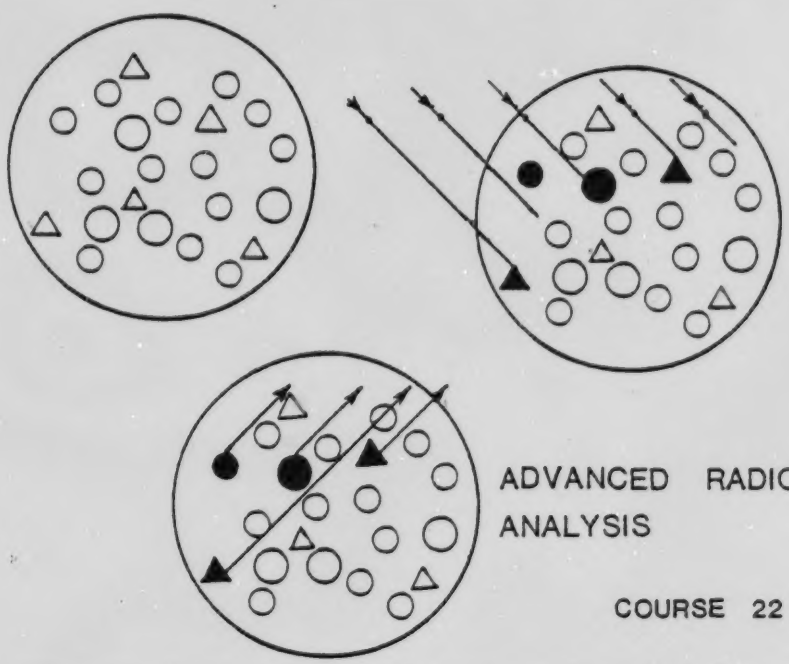
2. The procedure used to prevent adsorption of radionuclides in liquid samples is – _____
_____
_____

3. The following data should be included in a sampling notebook: _____
_____
_____
_____
_____
_____

NUCLEAR TECHNOLOGY

MODULE 22-2

GAMMA SPECTROMETRY



ADVANCED RADIONUCLIDE ANALYSIS

COURSE 22

CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 22 ADVANCED RADIONUCLIDE ANALYSIS

MODULE 22-1	Survey of Methods Used in Radionuclide Analysis (Sample Collection & Analysis)
MODULE 22-2	Gamma Spectrometry
MODULE 22-3	Alpha Spectrometry
MODULE 22-4	Beta Spectrometry
MODULE 22-5	Radiochemical Methods of Analysis by Liquid Scintillation Counting
MODULE 22-6	Neutron Activation Analysis
MODULE 22-7	Determination of Specific Nuclides (In the Environment)

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INTRODUCTION

In the nuclear field, the term "spectrometry" refers to the use of a spectrometer to measure the energy distribution of a particular type of radiation. Although an energy analysis can be performed for any type of radiation, gamma analysis is of greater significance because it has become the principal method of nuclide determination. Unknown radionuclides are identified and sorted by the spectrometer.

Gamma spectrometry is important for several other reasons: (1) Gamma radiation is an extremely penetrating and harmful type whose detection and analysis are critical. (2) The equipment required is relatively simple but quite efficient. (3) Spectrometry is employed in the detection of X-ray emitters.

This module discusses gamma radiation, gamma-ray detectors, and gamma-ray spectrum and their relationship to gamma spectrometry. Specific procedures for spectral analysis with a multichannel analyzer are provided.

A Gamma Ray Energy Table such as the one by Filby is required for this module. (Refer to "Reference Materials" section of this module.)

MODULE PREREQUISITES

The student should have completed a course in basic algebra and Course 11, "Radiation Detection and Measurement."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Define the following terms:
   - Gamma-ray spectrum.
   - Rayleigh scattering.
   - Thompson scattering.
   - Photoelectric effect.
   - Compton scattering.
   - Pair production.
   - Single- and double-escape peak.
   - Compton edge.
   - Resolution.
   - Full width at half maximum.
   - Spectral stripping.
   - Specific activity.
2. List and describe the three major interactions by which gamma rays lose energy in matter.
3. Discuss the relative advantages and disadvantages of the two major types of gamma-ray detectors.
4. Calculate the efficiency of a detector given the observed counting rate and known disintegration rate of a radionuclide.
5. List the four components of a gamma-ray spectrum.
6. Describe the procedure used to strip the gamma-ray spectrum of a mixture of one known and one unknown gamma-ray emitting radionuclide.
7. Given a known source and a gamma-ray spectrometer, calibrate the instrument, and determine its counting efficiency.

8. Use a gamma-ray spectrometer to identify an unknown radionuclide and to determine its disintegration rate.

GAMMA SPECTROSCOPY

DEFINITIONS AND PRINCIPLES

Gamma rays originate in the nuclei of atoms. Such rays are created when the nucleus of a radioactive atom emits an alpha or beta particle and the daughter nucleus is left in an excited state. The excess energy usually is emitted as gamma radiation, bringing the nucleus to a more stable energy condition. Because gamma rays are the result of the transition from one exact energy state to another exact energy state, they are emitted as mono-energetic and predictable radiation. The term "mono-energetic" radiation refers to the particular and different energy emitted by each source.

This emission characteristic of gamma rays makes gamma spectroscopy useful in the identification of a radionuclide that emits gamma radiation. Gamma spectroscopy allows the technician to sort gamma rays by energy. Since the rays are mono-energetic, an exact determination of their energy will help identify the radionuclide from which they are emitted. Gamma-ray spectroscopy is the determination of the energy distribution of gamma rays emitted by nuclei.

Gamma-ray detectors take advantage of the way in which gamma rays interact with matter to create a pulse of electrons that has a pulse height directly proportional to the energy of the gamma ray that created the pulse. This process produces a display of counter pulses that result from gamma rays as the function of their energy; the display is

called a "gamma-ray spectrum." This spectrum is a two-dimensional display. The energy of the gamma rays usually is displayed horizontally, and the number of counts at any particular energy is displayed vertically. Figure 22-2-1 depicts a typical gamma-ray spectrum taken from a trace recorder.

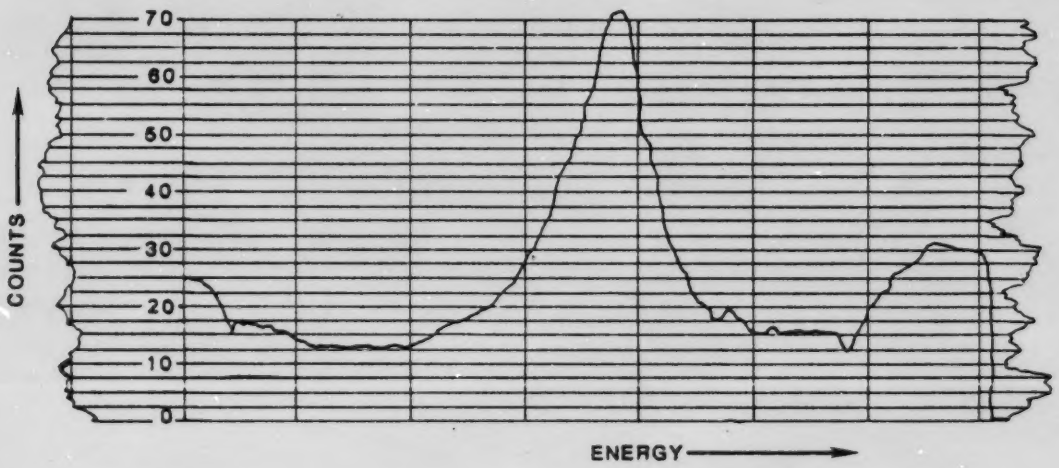


Figure 22-2-1. Gamma-ray spectrum, ^{137}Cs.

GAMMA RADIATION: INTERACTIONS WITH MATTER

Gamma radiation can interact with nuclei, with fields around nuclei, or with orbital electrons, although interactions with electrons are most common. There are five kinds of gamma-ray interaction with orbital electrons:

- Rayleigh scattering.
- Thompson scattering.
- The photoelectric effect.
- The Compton effect.
- Pair production.

Gamma rays that lack the energy to cause excitation or ionization often cause an effect called "Rayleigh scattering." In this interaction, the gamma ray collides with an electron and "bounces" off. The energy of the gamma ray is unchanged (Figure 22-2-2).

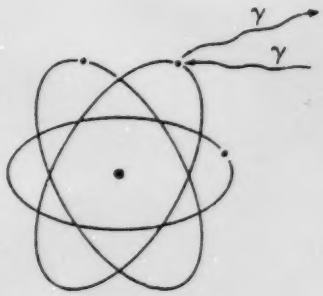


Figure 22-2-2.
Rayleigh scattering.

Slightly more energetic gamma rays can cause Thompson scattering (Figure 22-2-3). The electron absorbs the gamma ray, oscillates in an excited state, and then radiates a gamma ray in a random direction.

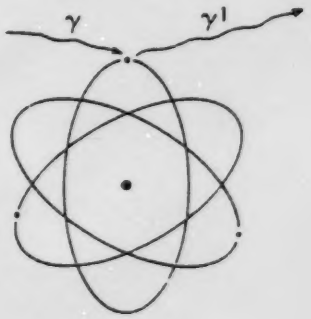


Figure 22-2-3.
Thompson scattering.

Interactions by which the energy level of an atom is changed are most important in gamma-ray spectroscopy because the energy of the gamma-ray is transferred to the detector atoms. In the photoelectric effect (Figure 22-2-4), gamma-ray energy is absorbed by an electron, and the

electron is knocked out of its orbit to form an ion pair. The electron leaves the atom with an energy equal to the energy of the incoming gamma ray minus the electron binding energy. All the energy of the gamma ray is transferred to the electron.

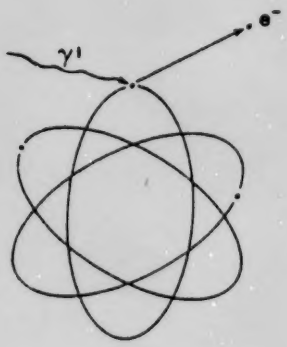


Figure 22-2-4.
The photoelectric effect.

Gamma rays also can lose energy to an electron by Compton scattering (Figure 22-2-5). This interaction is similar to the photoelectric effect, except that all the energy of the gamma ray is not given to the electron. As the electron is ejected from its orbit, another gamma ray leaves the atom at a slightly reduced energy. The energy of the new gamma ray is equal to the energy of the old gamma ray minus the kinetic energy and former binding energy of the electron.

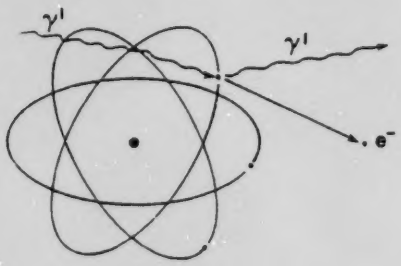


Figure 22-2-5.
Compton effect.

In the general region between 500 keV and 2 MeV, where most gamma rays occur, Compton scattering is the predominant mode of losing energy; and, as a result, there are many Compton-scattered gamma rays in these regions. Their energies commonly range from the initial energy minus 300 keV to zero. Compton scattering provides a major source of interference in the gamma spectrum. Gamma-ray spectra show a sharp increase in Compton-scattered gamma rays approximately 300 keV below the primary gamma-ray energy. This increase is the Compton edge.

At very high energies (greater than 1.02 MeV), gamma rays can interact with the electromagnetic field of the nucleus. When this occurs, a pair of particles – an electron and a positron – is created. This process is called pair production (Figure 22-2-6). The electron and the positron fly off into space, where they lose energy by collisional de-excitation. If the positron encounters an electron, it will be annihilated. The annihilation of this positron and electron will create two 0.511-MeV gamma rays.

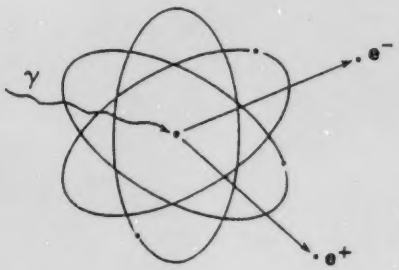


Figure 22-2-6.
Pair production.

If one of the two 0.511-MeV gamma-rays resulting from the positron annihilation escapes from the detector, the energy of the gamma ray that the detector "sees" is equal

to the original energy of the gamma ray minus the escaping 0.511 MeV energy, or —

$$E_\gamma \text{ (Detector sees)} = E_\gamma \text{ (original)} - 0.511 \text{ MeV}.$$

The peak which results is called a "single escape peak." The term "double escape peak" applies when both the 0.511-MeV gamma rays escape:

$$E_\gamma \text{ (Detector sees)} = E_\gamma \text{ (original)} - 2 \times 0.511 \text{ MeV}.$$

GAMMA-RAY DETECTORS

Currently, two principal types of gamma-ray detectors are used:

- The sodium iodide crystal detector.
- The lithium-drifted germanium detector.

The principles of both detectors have been discussed thoroughly in Course 11, Module 5, of this series.

Figure 22-2-7 is a sketch of a sodium iodide scintillation detector and photomultiplier tube. When gamma rays enter the sodium iodide, light is created. The photomultiplier tube, located directly behind the sodium iodide detector, converts this light into electrons that create an electronic pulse. The amount of light created is directly proportional to the energy of the gamma rays. There is a direct correlation between the amount of light and the number of electrons at the end of the photomultiplier tube.

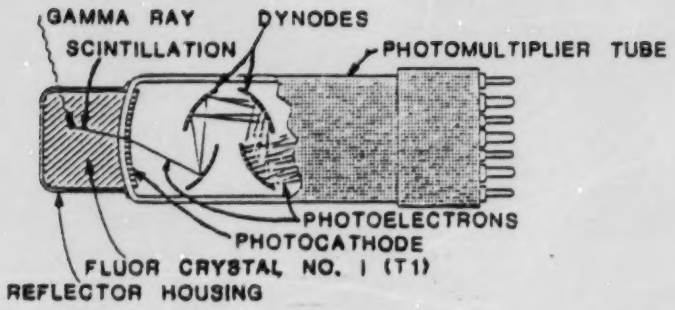


Figure 22-2-7. Sodium iodide scintillation detector and photomultiplier tube.

The lithium-drifted germanium detector is a semiconductor detector. Gamma rays create a short path of ionized germanium-lithium atoms that are withdrawn to one side of the detector, where they create a pulse directly proportional to the energy of the gamma ray. This detector, written GeLi and pronounced "jelly," is operated at very low temperatures and must be kept cold with liquid nitrogen.

Each of these detectors possesses unique advantages and operating characteristics. Both types differ in resolution and efficiency. Resolution is the ability of the detector to separate two gamma rays of equal energies. The unit of resolution is the full width at half maximum, which represents a peak of a general Gaussian shape measured by its width at half maximum (Figure 22-2-8). Points A and B represent different energies at full width and half maximum. If a detector has a band width similar to the width of the curve depicted in Figure 22-2-8, it will not be able to distinguish between the energies represented by points A and B; and its resolution will be very poor.

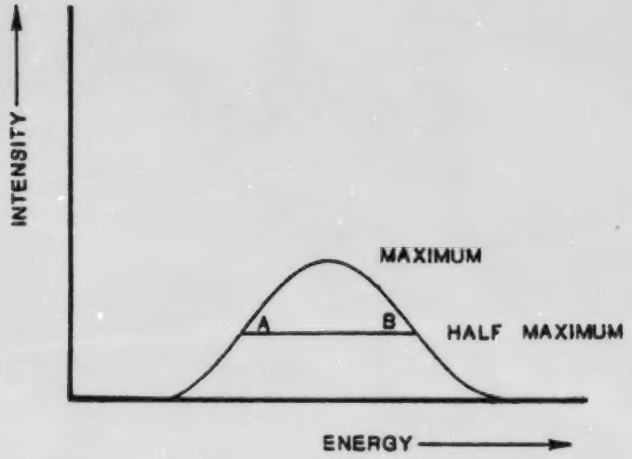


Figure 22-2-8. Full width at half maximum of spectra taken on sodium iodide detector.

If a detector has a full width at half maximum, as depicted in Figure 22-2-9, its resolution will be considered rather good, since it will be able to distinguish and resolve two gamma rays of approximately equal energy.

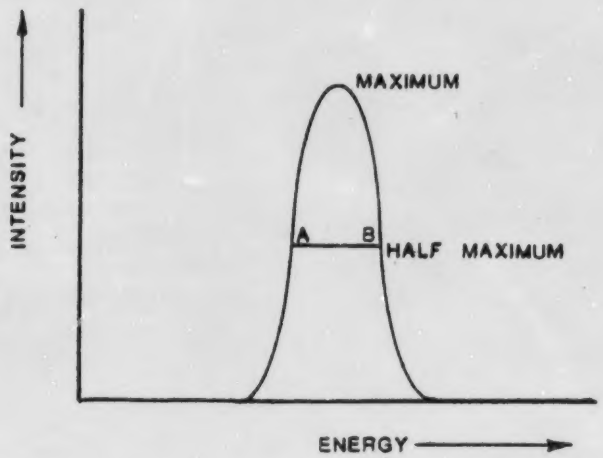


Figure 22-2-9. Full width at half maximum of spectra taken on GeLi detector.

High quality GeLi detectors possess this kind of resolution and have about 25 times the resolution of a high-quality sodium detector. These detectors are the best choice for the technician who needs a high-resolution detector to determine the presence of gamma rays that have very similar energies.

Efficiency of a detector can be expressed by the following equation:

$$\% \text{ efficiency} = \frac{\text{observed counting rate}}{\text{known disintegration rate}} \times 100 \qquad \text{Equation 22-2-1 (Equation = Eq.)}$$

Efficiency is a measurement of the pulses per unit time delivered by a detector compared to the incident number of gamma rays emitted to that detector.

Although the GeLi detector possesses superior resolution, the sodium iodide detector is more efficient; in fact, the efficiency of GeLi detectors often is stated as a percentage of the efficiency of a sodium iodide crystal. This means that a "15% efficient" GeLi detector detects 15% of the gamma rays that a sodium iodide crystal would detect. Obviously, when a low count rate is anticipated and high efficiency is desired, the proper choice is the sodium iodide detector. This detector functions with efficiencies that range from 6 to 30 times greater than the efficiencies of GeLi detectors.

Figure 22-2-10 illustrates the advantages and disadvantages of each detector type. This figure is a graph of complex gamma spectrums of ^{166}Ho obtained with a GeLi detector. The sodium iodide detector was unable to resolve

the complex spectrum, although its counting efficiency was much greater. But the clear, sharp peaks of the GeLi detector almost completely characterize the spectrum.

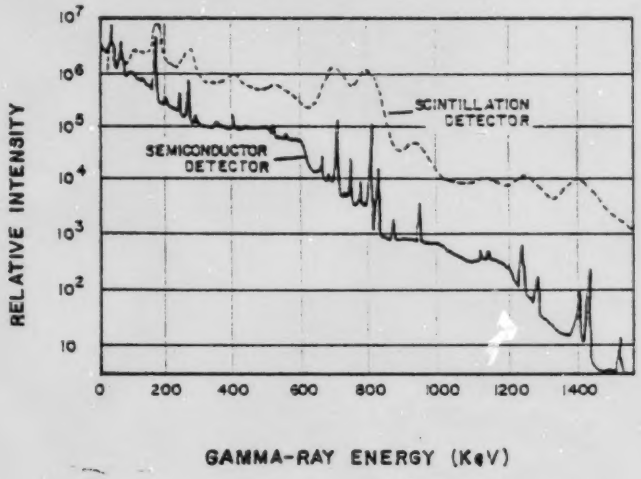


Figure 22-2-10. Complex spectrum of ^{166}Ho.

The GeLi detector possesses one other advantage that makes it far more valuable than the sodium iodide detector for the identification of individual radionuclides:

- The ability to resolve different closely-grouped gamma-ray energy peaks.

This capacity is an important one because a gamma-ray spectrum consists of four different components – all of which are added together to make the final spectrum.

GAMMA-RAY SPECTRUM

The four components of the gamma-ray spectrum are –

- The background.
- The gamma rays caused by the radionuclide.
- The Compton edges from each gamma ray.
- The single- and double-escape gamma rays.

All of these gamma rays obviously confuse the situation. They must be sorted out and their influences taken into account before isolation of the energy produced by the radionuclide and identification of the radionuclide. This analysis is complicated, but is made easier by the multichannel analyzer.

MULTICHANNEL ANALYZER

The pulses from a gamma-ray detector are fed into a multichannel analyzer (MCA). Figure 22-2-11 is a block diagram of an MCA in its simplest form.

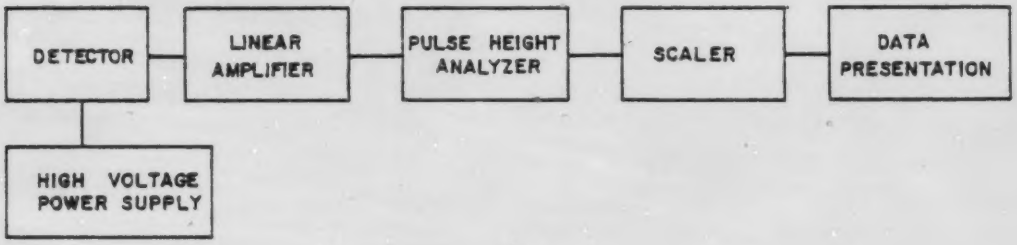


Figure 22-2-11. Multichannel analyzer spectroscopy system.

The signal from the detector is fed through an amplifier and then into a pulse-height analyzer. There, the signal is sorted by size and assigned to one of the many channels, or accumulation units. Each channel allows the passage of a pulse having a height in a specific range. Each channel, or analyzer, feeds a separate scaler, and each scaler accumulates counts for a specific pulse-height range.

Since pulse height is directly proportional to the energy of the gamma ray that causes the pulse, the pulse height can be calibrated in units of energy. A typical expression of pulse-height analyzer calibration, or output, is in units of energy (keV) per channel. A simple 400 channel analyzer, covering the range of 0 to 2 MeV, would sort pulses into channels in units of 2000 keV/400 channels = 5 keV/channel. Some MCAs in general use contain 1024 or 2048 channels. Because of the method of sorting pulses by pulse height, spectroscopy also is known as "pulse-height analysis."

Signals from the pulse-height analyzer drive scalers. Data accumulated in scalers can be presented in many ways — directly on an oscilloscope screen, on a x-y plotter that will draw the spectrum on a graph, or on a printout from an electric or Franklin typewriter that will print out channel number and counts in each channel. Information contained in a pulse-height analyzer can be placed onto magnetic tapes, computer discs, or punched paper tapes. In these forms, data can be stored or processed through a computer that can analyze the spectra. Many MCAs, even the small ones, now are computer based.

SPECTRAL ANALYSIS

The four components of the gamma-ray spectrum can be isolated by a procedure known as "spectral stripping." Components of the spectrum thereby are separated and isolated by the gamma-emitting radionuclides.

If one component of a radionuclide mixture is known, the spectrum of the known component can be stripped by hand by use of the MCA. Some multichannel analyzers strip the component electronically.

CALIBRATION OF THE GAMMA SPECTROMETER

Radioactive sources are used to calibrate the spectrometer. All sources used must meet these criteria:

- Use well-characterized radionuclides.
- Have long half-lives.
- Have a known activity.

The U.S. National Bureau of Standards provides a source often used – a mixed gamma-ray source that contains approximately 8 gamma rays having energies between 0 and 2 MeV.

With a good quality source available, the technician calibrates the detector by following these steps:

- The source is counted.
- The energy of the gamma ray (obtained from references) is plotted against the channel number.
- A straight line is drawn between the points.

The straight line will coordinate the energy of the gamma ray with the appropriate channel numbers.

EXAMPLE	
Given:	Known sources of gamma rays, such as ^{137}Cs or ^{60}Co, and a spectrometer of the type illustrated in Figure 22-2-12.
Find:	Energy calibration curve for the spectrometer.
Solution:	Set up the MCA spectroscopy system as in Figure 22-2-11. A typical MCA analyzer with CRT display is depicted in Figure 22-2-12. Adjust the "AMP Gain" to obtain the optimum count. As an example, if a ^{60}Co source is used, its highest

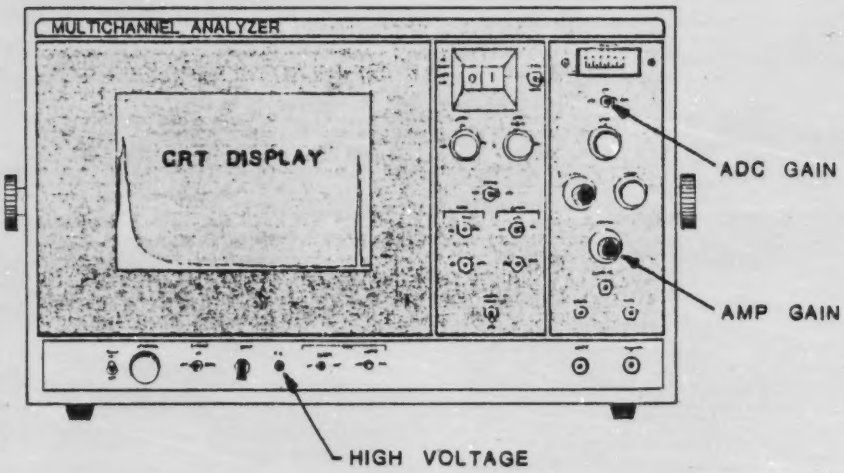


Figure 22-2-12. Typical modern multichannel analyzer.

energy gamma-ray pulse (1.33 MeV) should be adjusted on the screen in such a manner that the peak to this gamma ray is found approximately halfway across the spectrum. The channel number of a given gamma-ray energy can be increased by adjustment of the knobs marked "AMP Gain," "ADC Gain," or of the "HV" (high voltage) to a NaI detector. CAUTION: At no time should the high voltage be adjusted to an amount greater than that recommended by the instructor; however, the "ADC Gain" or "AMP Gain" can be adjusted to any value desired without harm to the instrument.

Once the "HV" and "ADC Gain" and "AMP Gain" knobs have been selected and the calibration procedures started, none of them should be touched again without recalibration. Recalibration is accomplished by the counting of at least two different gamma rays; for example, ^{137}Cs (E_γ = 0.66

MeV) and a ^{60}Co (E_γ = 1.17 MeV and 1.33 MeV) can be used.

Each source should be counted separately, and the channel number in which the peak of the gamma ray is found should be ascertained by scanning across the addresses and by using the numeric display of channel count. A table similar to Table 22-2-1 is completed; the channel number for each gamma-ray energy is used, and the energy of the gamma ray listed.

TABLE 22-2-1

Channel Number	Gamma Ray Energy (MeV)
110	0.661
190	1.17
210	1.33

From the Table 22-2-1 data, a graph is plotted for the channel number on the y-axis versus the gamma energy on the x-axis. A linear relationship is obtained in the form illustrated in Figure 22-2-13. Use this figure to determine the gamma-ray energy of an unknown nuclide whose peak channel is known; for example, a peak channel of 150 has a corresponding energy of about 0.8 MeV.

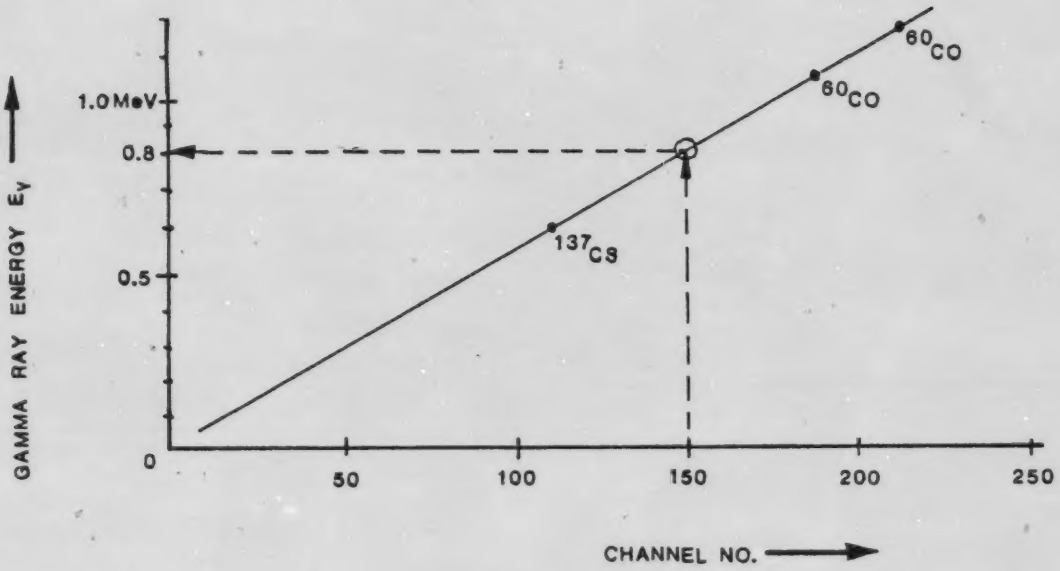


Figure 22-2-13. Energy calibration curve.

Sometimes the counting efficiency of a detector must be determined. Since the closer the source, the more efficient the detector, efficiency is a function of geometry and the energy of the gamma. Efficiency can be determined if –

- The source in a known geometry is counted.
- The counts per minute for a given gamma ray is compared to the number of rays per minute emitted by the source (the standard).

Efficiency then can be calculated from Equation 22-2-1. The number of gamma rays per minute emitted by the standard is obtained from its activity data. An efficiency curve for each counting geometry can be obtained if efficiency versus the energy of the gamma ray is plotted on log-log paper, as, for example, in Figure 22-2-14.

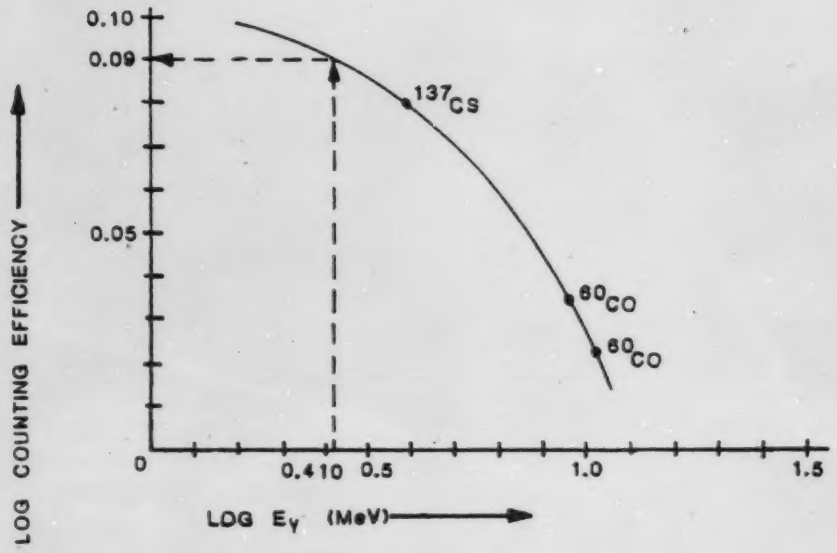


Figure 22-2-14. Typical count efficiency curve.

IDENTIFICATION OF RADIONUCLIDES

Once the instrument is calibrated, the technician can identify unknown nuclides with a spectrometer by the following procedure:

- Count unknown nuclide with gamma spectrometer.
- Identify each gamma-ray peak displayed.
- Use MCA to find peak channel and net count per minute under each peak for peak ratio and $T_{\frac{1}{2}}$ determinations.
- Calculate energy of gamma ray from energy calibration curve.
- Compare energy of gamma to chart or reference table that lists characteristic energies for radionuclides. In this way, the radionuclides present can be identified.

EXAMPLE

Given: (1) A calibrated MCA and a calibration curve (Figure 22-2-13). (2) Reference tables given by Filby, Weast, or similar ones (refer to "Reference Materials" section in this module).

Find: Unknown radionuclide.

Solution: By counting the unknown nuclide and discovering that it has one gamma ray found in channel number 80, we determine this gamma-ray energy to be 0.410 MeV. From reference tables, determine that a radionuclide that has one gamma ray with an energy of 0.410 MeV is ^{198}Au. This is a reasonable choice for the following reasons:

- Neutron capture cross section for ^{197}Au to ^{198}Au is very large.
- ^{198}Au has one gamma ray 0.410 MeV.

The initial radionuclide determination can be double-checked by counting the radioisotope over several weeks, with the expectation of finding a disintegration rate that follows the 2.6-day half-life of ^{198}Au.

Once the radionuclide has been identified, its disintegration rate can be determined as follows:

- Obtain a count rate of each gamma ray in counts per second (cps).
- Determine branching ratio from the tables.

Having made an energy calibration for the counting efficiency of a detector, plot the counting efficiency on log-log paper to obtain the efficiency curve. Use the counting effi-

ciency curve to determine the counting efficiency of the radionuclide in question, and calculate the disintegration rate from Equation 22-2-2.

$$\frac{cps}{BR\ (E)} = dps \qquad \text{Eq. 22-2-2}$$

where:

cps = counts per second.
BR = branching ratio.
E = counting efficiency.
dps = disintegrations per second.

EXAMPLE	
Given:	^{198}Au radionuclide has 9000 count/100 secs. Set of reference tables, as in previous Example.
Find:	Disintegration rate of unknown sample.
Solution:	From the reference tables, the 0.410-MeV gamma ray has a branching ratio of 1.0 (or 1 gamma ray for each disintegration). From the counting efficiency curve (Figure 22-2-14), note that a gamma ray of 0.410 MeV has a counting efficiency of 0.09, which means that there are nine counts for every 100 gamma rays emitted by the radionuclide. Calculation from Equation 22-2-2 yields the following results: $cps = \frac{9000}{100} = 90$ cps $BR = 1.0$

$$E = 0.090$$

$$\therefore \text{dps} = \frac{90}{1.0 \times 0.090}$$

$$= 1000 \text{ dps.}$$

The number of microcuries is 1000 dps/3.7 x 10^4 dps/μCi = 0.03 μCi. This value means that the unknown radionuclide has been identified as gold 198 and that the sample contains 0.03 μCi of radioactivity.

EXERCISES

(NOTE: A table that relates channel numbers to energy is required for these exercises; one such table is that by Filby, referred to in the "Reference Materials" section of this module.)

1. Determine the energy of a gamma found in channel #1285. The 1.17-MeV and the 1.33-MeV peaks of the ^{60}Co are in channel 1000 and 2000, respectively.
2. The nuclide ^{137}Cs decays 100% of the time with a 0.667 MeV gamma. If efficiency of a 0.667 MeV gamma = 25%, what is the dpm of a 1000 cpm source of ^{137}Co? How many μCi are there if 1 Ci = 3.7 x 10^{10} dps?
3. Given the following spectral data, determine the peak channel and the counts in the peak.

	1	2	3	4	5	6	7	8	9	10
0	000102	000084	000072	000076	000076	000077	000082	000089	000075	000079
10	000072	000080	000057	000065	000079	000077	000087	000089	000080	000079
20	000086	000091	000105	000210	000814	002816	004247	002219	000396	000062
30	000045	000052	000053	000053	000058	000060	000047	000045	000049	000057

MATERIALS

Source ^{137}Cs, ^{22}Na, ^{60}Co, ^{89}Y, and unknown radionuclide.
Detector – NaI or GeLi.
Multichannel analyzer with output.
Reference book that contains gamma energies. (The tables given by Filby or Weast are adequate; refer to "Reference Materials" section of this module.)

PROCEDURES

1. Set up equipment and calibrate energy.
   a. Arrange as in Figure 22-2-11.
   b. Calibrate E_γ vs. channel number.
      (1) Count several standards.
      (2) Plot E_γ vs. channel number, as illustrated in Figure 22-2-13.
      (3) Draw straight line.
2. Calibrate efficiency:
   a. Count standard source with known activity.
   b. Determine the cps in each γ.
   c. Using the source strength and branching ratios, obtain γ's per sec for each γ.
   d. Calculate E = cps/γ's per sec.
   e. On log-log scale, plot E vs. E_γ; draw a smooth curve.
3. Count unknown: identify and determine activity.
   a. Count.
   b. List channel number and counts/sec.

c. Determine E_γ from the energy calibration curve.

d. From E_γ and the first reference, determine the nuclide.

e. From cpm get γ's/sec from the efficiency curve.

f. From any of the first three references, obtain the branching ratios; and transform γ's per sec to dps for each γ, using Equation 22-2-1.

g. From the average dps values for each nuclide, determine activity in curies. 1 Ci = 3.7×10^{10} dps.

DATA TABLE 22-2-1

ENERGY CALIBRATION DATA

E_γ	Channel No.

DATA TABLE 22-2-2

COUNTING EFFICIENCY CALIBRATION DATA

E_γ	cps	γ's ps	$\frac{\text{cpm}}{\gamma\text{'s ps}} = E$

DATA TABLE 22-2-3

IDENTIFICATION OF UNKNOWN NUCLIDE AND DETERMINATION OF DISINTEGRATION RATE

Peak Channel, E	Area in Peak, cps	dps in peak	Activity	Nuclide

REFERENCE MATERIALS

Chase, Grafton D. and Rabinowitz, Joseph L. Principles of Radioisotope Methodology. 3rd ed. Minneapolis: Burgess Publishing Company, 1967.

Filby, R. H., et al. Gamma Ray Energy Table for Neutron Activation Analysis. Report No. WSUNRC-97-2. Pullman, WA: Washington State University, 1970.

Lederer, C. M.; Hollander, J. M.; and Perlman, I. Table of Isotopes. 6th ed. New York: John Wiley and Sons, Inc., 1968.

Weast, Robert C., ed. CRC Handbook of Chemistry and Physics. 55th ed. Cleveland, OH: CRC Press, 1974. (This work is updated yearly.)

Wang, C. H.; Willis, D. H.; and Loveland, W. D. Radiotracer Methodology in the Biological, Environmental and Physical Sciences. Englewood Cliffs, NJ: Prentice-Hall, 1975.

1. Define the following terms:
   a. Gamma-ray spectrum.

   b. Rayleigh and Thompson scattering.

   c. Compton edge and Compton scattering.

   d. Escape peak.

   e. Full width at half maximum.

   f. Resolution.

   g. Photoelectric effect.

h. Spectral stripping.

i. Pair production.

j. Specific activity.

2. Describe the three major ways in which gamma rays lose energy in matter.

3. Name two types of gamma-ray detectors. Describe the advantages and disadvantages of each.

4. List the four components of a gamma-ray spectrum.

5. Why is spectrum stripping necessary in the analysis of a gamma-ray spectra?

6. A count on a MCA shows 2 peaks with the following E_γ's and cpm. Identify the radionuclide source.

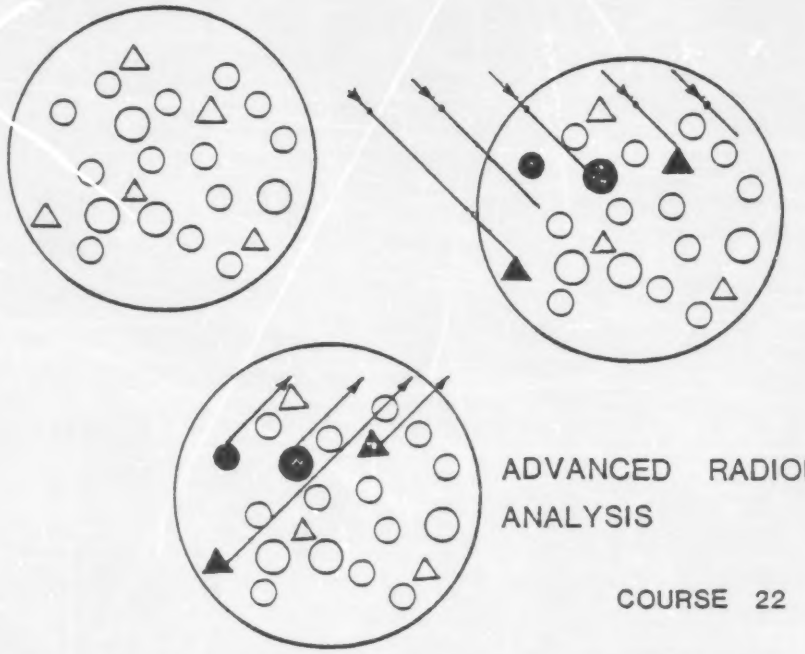
E_γ	cpm
1.17 MeV	1100
1.33 MeV	1000

7. List all the possible observed peaks from a source that emits only a 2.73-MeV gamma.

NUCLEAR TECHNOLOGY

MODULE 22-3

ALPHA SPECTROMETRY



ADVANCED RADIONUCLIDE ANALYSIS

COURSE 22

ORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 22 ADVANCED RADIONUCLIDE ANALYSIS

MODULE 22-1	Survey of Methods Used in Radionuclide Analysis (Sample Collection & Analysis)
MODULE 22-2	Gamma Spectrometry
MODULE 22-3	Alpha Spectrometry
MODULE 22-4	Beta Spectrometry
MODULE 22-5	Radiochemical Methods of Analysis by Liquid Scintillation Counting
MODULE 22-6	Neutron Activation Analysis
MODULE 22-7	Determination of Specific Nuclides (In the Environment)

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INTRODUCTION

The techniques and principles of alpha spectroscopy are similar to those used to obtain and analyze gamma spectra. The major differences are the characteristics of the spectra and the detectors used.

This module briefly discusses alpha radiation, alpha-particle decay, and the relationship of these phenomena to alpha spectroscopy. Specific procedures for identification of an unknown alpha-emitting radionuclide are presented.

MODULE PREREQUISITES

The student should have completed a course in basic algebra and Course 11, "Radiation Detection and Measurement."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Describe alpha decay and alpha emission.
2. Describe the "energy-well concept" and its relationship to half-life, atomic number, and alpha energy.
3. Describe a Bragg curve and its relationship to specific ionization and range.
4. List the two major types of alpha detectors, and describe the ways in which they are used for alpha spectroscopy.
5. Detail the procedures employed in the identification of an unknown alpha-emitting radionuclide.

6. Explain how the technician can identify the atomic mass, number, and half-life of a nuclide if its alpha-emitting energy is known.
7. Mount, count, and identify an unknown alpha-emitting radionuclide.

ALPHA PARTICLES

Alpha particles are ionized helium nuclei ejected during the disintegrations of radioactive nuclei of various heavy elements. They are positively-charged particles that consist of two protons and two neutrons. The particles are emitted from radioactive materials at speeds that range from 1.6×10^5 to 1.6×10^6 cm/sec. Because of their size and relatively slow speeds, their range is short; and they travel only a few (2-8) centimeters in air before being stopped.

Alpha-particle decay and emission occur in the heaviest natural elements and in the transuranic elements, specifically in those elements that have a mass greater than 200 AMU and an atomic number greater than 83. An alpha particle is structurally the same as a ^4_2He particle; and since helium is an element that undergoes alpha decay, it changes its mass by 4 and its atomic number by 2. ^{238}U, with 92 protons, will decay to ^{234}Th with 90 protons. Some alpha emitters are ^{238}U, ^{236}U, ^{232}Th, their daughters, and the man-made transuranic nuclides, such as ^{239}Pu and ^{240}Pu.

Alpha-particle emission is the removal of excess mass from nuclei that simply are "too large." The emitted particle must have enough energy to overcome the barrier of positively-charged nucleons at the surface of the nucleus. The larger the energy of the alpha particle, the easier it can overcome the barrier. Figure 22-3-1 consists of a diagram of this nuclear potential barrier. The barrier is represented by the curves; the inside of the nucleus is depicted as an energy well from which the alpha particle must escape. The particle can go over or through the barrier.

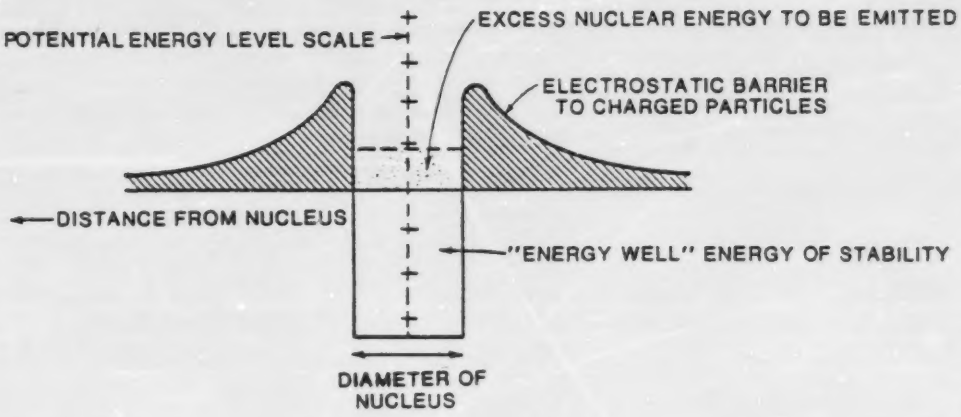
POTENTIAL ENERGY LEVEL SCALE
EXCESS NUCLEAR ENERGY TO BE EMITTED
ELECTROSTATIC BARRIER TO CHARGED PARTICLES
DISTANCE FROM NUCLEUS
"ENERGY WELL" ENERGY OF STABILITY
DIAMETER OF NUCLEUS

Figure 22-3-1. Diagram of the nuclear potential barrier and "energy-well" concept.

The "energy-well" concept helps to explain why that, for a given iostope, half-life becomes shorter as alpha-particle energy increases, since high energies allow the particles to escape from the well and, therefore, to lower the half-life of the isotope. Nuclides that decay with relatively low alpha-particle energies have a very long half-life because a relatively large number of attempts are required for the particle to escape over and through the potential barrier.

A definite relationship exists between alpha-particle energy and atomic number: the higher the atomic number of a nuclide, the higher the alpha energy. Again, the energy-well concept helps to explain this relationship since the more protons there are in the nucleus, the higher the energy well and the larger the alpha-particle energy must be to allow escape.

Alpha particles have a characteristic and finite range. As an alpha particle slows down in an absorber, it creates a large number of ions at the very end of its path. This

increase of ionization at the end of a charged particle track is called the "Bragg effect" and has been used in the medical treatment of deep-seated tumors. Charged particles create only a few ions near the skin and a large number in the tumor, allowing the physician to destroy the tumor but to leave the skin relatively unaffected.

A plot of the ions created per millimeter in an absorber against the remaining range is called a "Bragg curve." Figure 22-3-2 is a Bragg curve for alpha particles that initially have the same energies.

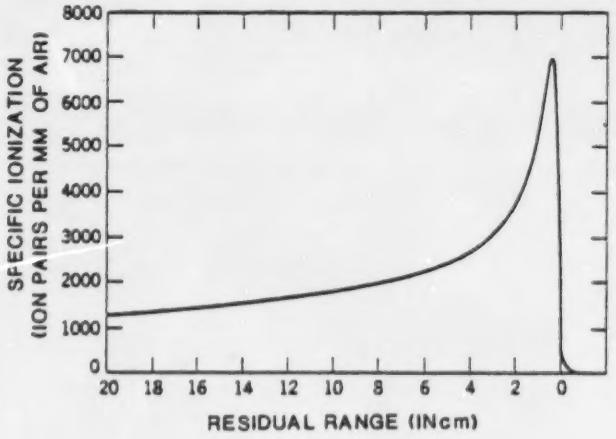

Figure 22-3-2. Bragg curve for monoenergetic alpha particles.

Alpha particles are emitted monoenergetically, although initially all possess the same energy. Each radionuclide emits alphas that possess characteristic energies. Thus, alpha detectors that produce an electronic pulse whose height is proportional to the energy of the radiation that caused the pulse can be used to identify unknown alpha-emitting radionuclides. In this sense, alpha spectroscopy is similar to gamma spectroscopy.

A radionuclide cannot be identified positively in every case by alpha energy alone because different nuclides may emit alpha particles of nearly identical energies. But, since alpha energy is related to half-life and to atomic number, once the half-life or atomic number is determined, a positive identification can be made.

ALPHA-PARTICLE DETECTION

Two types of detectors are in wide use for the detection of alpha particles: the gas proportional detector and the solid-state semiconductor detector.

Proportional counters are constructed in a variety of configurations, although the window and the windowless counters are the most popular ones. The operation of these detectors has been explained fully in Module 3 of Course 11, "Radiation Detection and Measurement." Because the alpha particle can be stopped by even very thin sheets of material, windowless counters often are utilized for alpha counting.

Solid semiconductor, lithium-drifted silicon (SiLi) detectors possess extremely good resolution compared to gas proportional counters. The operation of the semiconductor detector has been detailed in Module 5 of Course 11.

Both detectors are designed to create an electrical pulse directly proportional to the alpha-particle energy.

ALPHA SPECTROMETERS

The alpha spectrometer, like the gamma spectrometer, consists of a detector and of a counting system that amplifies

the pulses and sorts them according to height (energy). The signal from the detector is fed through an amplifier and then into a pulse-height analyzer. There, the signal is sorted by size and assigned to one of many available channels, or accumulation units. Each channel feeds a separate scaler, and each scaler accumulates counts for a specific pulse-height range. These pulses are calibrated in units of energy-per-channel and drive scalers that can present data in many ways. The system is the same as that for gamma spectroscopy, except an alpha detector is used rather than a gamma detector.

The process of radionuclide identification is similar. The spectrum of alpha-particle decay is plotted by correlation of the alpha energy and the channel number against the number of counts in each channel. Since there is a linear correlation between energy and channel number, the technician often can identify a radionuclide by determining the channel number associated with the maximum of a peak and the energy represented by that channel.

EXAMPLE	
Given:	Two common emitters, uranium-238 and americium-241.
Find:	The energy of an alpha particle whose peak appears in channel 100.
Solution:	Using the two given emitters, calibrate the alpha spectrometer. For ^{238}U, the 4.19-MeV alpha ray appears in Channel 30. The 5.48-MeV ^{241}Am alpha particle appears in Channel 110. Plot these values on a graph (Figure 22-3-3), with the x-axis as the channel number and y-axis as the

alpha energy. Draw a straight line joining the two points.

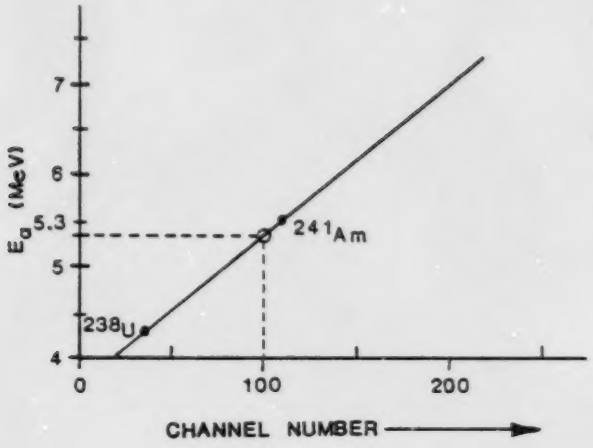

Figure 22-3-3. Energy calibration curve.

The alpha-particle energy now can be found if the channel number in which its signal appears is known; for example, an alpha particle whose peak appears in Channel 100 would have an energy of about 5.3 MeV.

Alpha spectra are easier to read than gamma spectra since there are no confusing Compton edges, double escape peaks, or scattered rays. A radionuclide, however, often cannot be identified by energy alone. Different nuclides may emit alpha particles of the same energy, in which case the particles' half-life and atomic number can be used to identify the nuclide by relating these quantities to the alpha energy.

Figures such as 22-3-4 and 22-3-5 often are employed instead of a complex equation to make this relationship and to determine the identity of a radionuclide. Figure 22-3-4 correlates decay energies and mass numbers for alpha emitters from astatine to fermium. Once the decay energy of a specific radionuclide is known, the isotope can be identified by the indexing of decay energy and of mass number for the element in question.

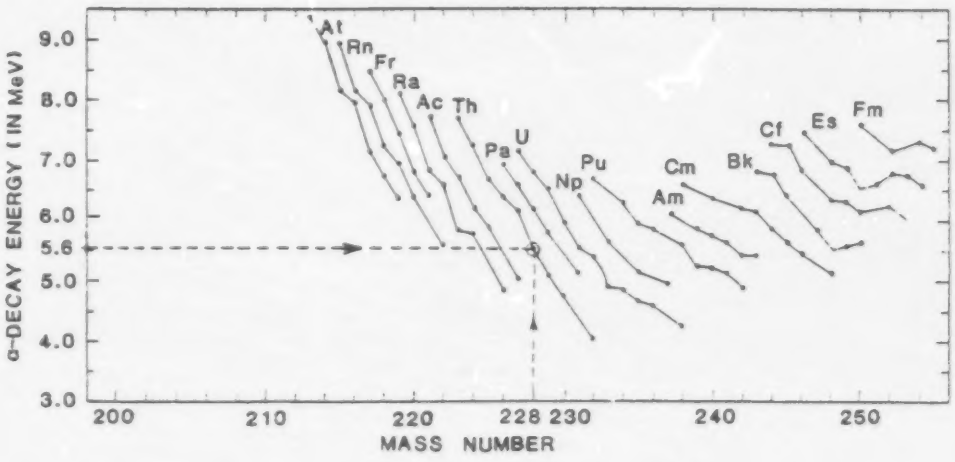

Figure 22-3-4. Plot of decay energies versus mass numbers for α emitters from astatine to fermium.

EXAMPLE	
Given:	An unknown thorium nuclide with an alpha decay energy of 6.25 MeV.
Find:	Identity of the nuclide.
Solution:	Use Figure 22-3-4. Find nuclide of thorium corresponding to an energy of 6.25 MeV. Read down to mass number. The nuclide is either ^{224}Ac or ^{227}Th.

Figure 22-3-5 can be used to determine the element undergoing disintegration when alpha energy and half-life are known.

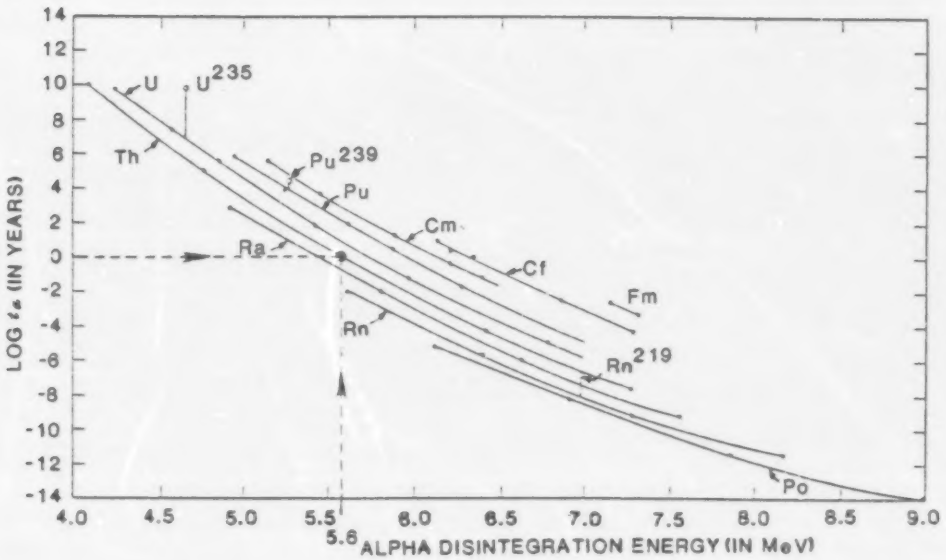

Figure 22-3-5. Relation between a partial α half-life and α-disintegration energy for even-even α emitters.

EXAMPLE	
Given:	An unknown alpha-emitting nuclide with an alpha-decay energy of 5.6 MeV and a half-life of 1.0 years.
Find:	Identity of the nuclide.
Solution:	From Figure 22-3-5, find the cross point corresponding to 5.6 MeV alpha disintegration energy and the log t_{α} = 0 (the log of 1.0 years = 0). This cross point comes closest to the line marked thorium; therefore, the alpha-emitting nuclide probably is an isotope of Th. Refer back to Figure 22-3-4, and note that since the alpha decay energy of 5.6 MeV was aligned closely with ^{228}Th, the radionuclide that has a count of 1.0 years half-life with a 5.6-MeV alpha-particle energy is identified as thorium 228. Note: Reference sources indicate that ^{228}Th exhibits a half-life of 1.9 years and an alpha-decay energy of 5.4 MeV.

This example demonstrates that, although use of Figures 22-3-4 and 22-3-5 allows approximate determinations, their use cannot provide exact identification. The x-axis in Figure 22-3-5 is the partial alpha-decay half-life, but is not necessarily the observed half-life. The observed half-life and the alpha-decay half-life are identical only if the radionuclide decays solely by alpha-particle emission. Figure 22-3-5 is limited further because it is valid only for even-even nuclides.

EXERCISES

1. What is the half-life of an unknown uranium nucleus having an alpha energy of 5.5 MeV?
2. Why does half-life decrease as alpha energy decreases?
3. An unknown nuclide of an element has an alpha energy of 6.0 MeV and a half-life of 36 days. What is the element?

MATERIALS

Multichannel analyzer with output.
Several known and unknown alpha sources.
Alpha-sensitive detector.
A table of isotopes. (Such a table is available from a number of sources; for example, from these two books listed in the "Reference Materials" section of this module: (1) CRC Handbook of Chemistry and Physics and (2) Table of Isotopes.)

PROCEDURES

IDENTIFICATION OF AN UNKNOWN ALPHA EMITTER

1. Calibrate equipment, using known alpha source.
2. Plot alpha energy against a channel number. Use Data Table 22-3-1, and plot curve in Figure 22-3-6.
3. Connect calibration point with line.
4. Count unknown source.
5. In Data Table 22-3-2, tabulate the channel numbers that correspond to the peaks of the alpha particles.
6. Determine alpha-particle energy from plot of energy calibration.

7. Instructor will provide either element identification or approximate half-life. Utilize Figures 22-3-4 and 22-3-5 and a table of isotopes (see references) to determine identity of radionuclide, mass number, and exact half-life. Complete Data Table 22-3-2.

DATA TABLES

DATA TABLE 22-3-1
KNOWN ALPHA SOURCE

E_α	Channel Number

DATA TABLE 22-3-2
UNKNOWN ALPHA SOURCE

Channel Number					
E_α					
Element Identification					
Approximate Half-life					
Radionuclide					
Mass Number					
Exact Half-life					

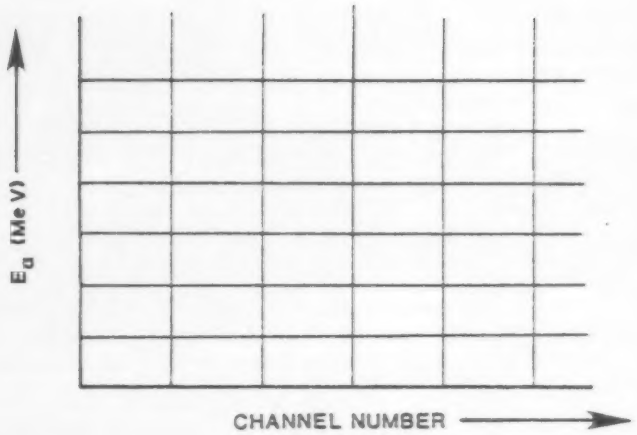

Figure 22-3-6. Energy calibration curve.

REFERENCE MATERIALS

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Friedlander, G.; Kennedy, Joseph W.; and Miller, Julian M. Nuclear and Radiochemistry. 2nd ed. New York: John Wiley and Sons, 1964.

Laderer, C.M.; Hollander, J.M.; and Perlman, I. Table of Isotopes. 6th ed. New York: John Wiley and Sons, 1967.

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Wang, C.H.; Willis, D.L.; and Loveland, W.D. Radiotracer Methodology in the Biological Environment and Physical Sciences. Englewood Cliffs, NJ: Prentice-Hall, 1975.

Weast, Robert C., ed. CRC Handbook of Chemistry and Physics. 60th ed. Cleveland, OH: CRC Press, 1979. (A new edition is published yearly.)

1. Describe alpha decay and alpha emission.

2. Describe the "energy-well concept" and its relationship to half-life, atomic number, and alpha energy.

3. Describe a Bragg curve and its relationship to specific ionization and range.

4. List the two major types of alpha detectors, and describe the ways in which they are utilized for alpha spectroscopy.

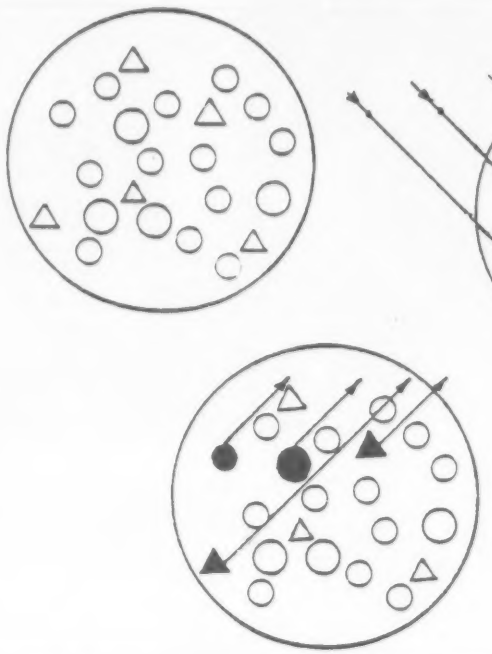
5. Detail the procedures employed in the identification of an unknown alpha-emitting radionuclide.

6. Explain how the technician can identify the atomic mass, number, and half-life of a nuclide if its alpha energy is known.

NUCLEAR TECHNOLOGY

MODULE 22-4

BETA SPECTROMETRY



ADVANCED RADIONUCLIDE ANALYSIS

COURSE 22

CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 22 ADVANCED RADIONUCLIDE ANALYSIS

MODULE 22-1	Survey of Methods Used in Radionuclide Analysis (Sample Collection & Analysis)
MODULE 22-2	Gamma Spectrometry
MODULE 22-3	Alpha Spectrometry
MODULE 22-4	Beta Spectrometry
MODULE 22-5	Radiochemical Methods of Analysis by Liquid Scintillation Counting
MODULE 22-6	Neutron Activation Analysis
MODULE 22-7	Determination of Specific Nuclides (In the Environment)

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INTRODUCTION

Although about 90% of all radionuclides undergo transformation by some form of beta-particle decay, beta analysis is performed infrequently to identify unknown nuclides. Beta analysis is a rather poor method for radionuclide identification because beta-particle energy is not monoenergetic and, therefore, nuclides cannot be sorted by differential energy spectra methods such as those employed in alpha and gamma spectroscopy. Other techniques of beta analysis must be used.

The most important and most practical of these techniques is Feather Analysis. This method allows the technician to determine beta-particle range, which then can be related to the nuclide's maximum beta-particle energy (written $E_{\beta max}$). $E_{\beta max}$ is the one unique feature of a radionuclide's beta decay that can be used to assist in the identification of the nuclide.

Beta analysis is used only when other forms of analysis (such as alpha or gamma spectroscopy) are impossible or impractical. These other methods are more accurate and should be utilized whenever possible.

MODULE PREREQUISITES

The student should have completed Course 11, "Radiation Detection and Measurements," and a course in basic algebra.

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Define and describe the following terms:
   Positron.
   Negatron.
   β^- emission.
   β^+ emission.
   K-capture.
   Isobar.
   Neutrino.
   $E_{\beta max}$.
   Magnetic spectrometer.
   Feather Analysis.
2. Write a sample decay scheme for β^- emission, β^+ emission, and K-capture.
3. Write an equation that defines the difference in energy between two isobars.
4. Use an energy distribution curve to determine $E_{\beta max}$ for a given nuclide, given its range in aluminum.
5. Explain why beta analysis is the least exact and least useful of the techniques for radionuclide identification.
6. Explain when beta analysis should be performed.
7. Describe the use of a magnetic spectrometer to sort beta particles by energy.
8. Describe the use of Feather Analysis to identify an unknown beta-emitting radionuclide.
9. Calculate the activity of an unknown nuclide, given transmission factors and activity of the nuclide at zero total absorber thickness.
10. Calculate apparent range, given absorber thickness and fraction of range.
11. With the Glendenin equation, calculate $E_{\beta max}$, given maximum range in mg/cm^2.

BETA PARTICLES

CHARACTERISTICS

Beta particles originate in the nucleus of an atom. Once outside the nucleus, they become high-speed electrons having an at-rest mass of about $\frac{1}{1837}$ that of a proton.

The term "beta particle" includes two differently-charged particles, positrons and negatrons. Positively-charged particles are called "positrons"; negatively-charged ones are called "negatrons." Although both the negatron and the positron are beta particles, the term "beta particle" refers only to the negatron. Except for their charge, both particles are physically alike, are formed by nuclear changes, and are ejected from the nucleus with velocities approaching the speed of light.

Beta particle (β^-) emission increases the atomic number of the residual nucleus by one, leaving the atomic mass essentially unchanged. The residual nucleus becomes the next highest element. This transformation is represented by Equation 22-4-1.

$$ {}^{A}_{Z}X \rightarrow {}^{A}_{Z}{}' + {}_{1}X + {}_{-1}e^{0} \qquad \text{Equation 22-4-1 (Equation = Eq.)} $$

In this equation, the beta particle is represented by ${}_{-1}e^{0}$.

This type of decay is typical for nuclei that possess a neutron excess. The electron is ejected from the nucleus; and the proton remains, raising the atomic number by one and leaving the mass number the same. Figure 22-4-1 illustrates the decay scheme for ${}^{32}_{15}P$, which is an example of

pure beta decay.

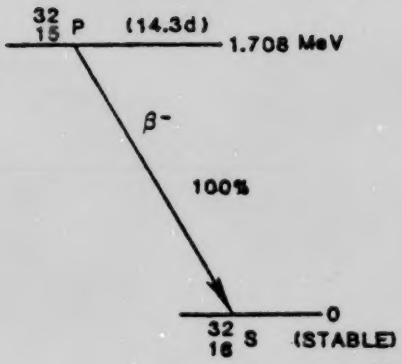


Figure 22-4-1.
Decay scheme for $^{32}_{15}P$.

In the positron (β^+) emission, the residual nucleus moves to an element that has a lower atomic number, and the atomic number is reduced by one. The atomic mass remains essentially unchanged. Equation 22-4-2 represents this transformation.

$$^{A}_{Z}X \rightarrow {}^{A}_{Z-1}X + {}_{+1}e^{0} \qquad \text{Eq. 22-4-2}$$

The positron is represented by ${}_{+1}e^{0}$. This type of decay is typical for nuclei that contain a proton excess (a nucleus that is neutron deficient). Positron emission actually occurs by the conversion of the excess protons into a positron and a neutron (Equation 22-4-3).

$$^{1}_{1}P \rightarrow {}^{0}_{+1}e + {}^{1}_{0}n \qquad \text{Eq. 22-4-3}$$

This reaction requires an excess energy of 1.02 MeV for completion; that is, the radioactive nucleus must have an energy of at least 1.02 MeV greater than the daughter nucleus in order that the reaction can take place. An example of B^+ decay of $^{14}_{8}O$ is illustrated in Figure 22-4-2.

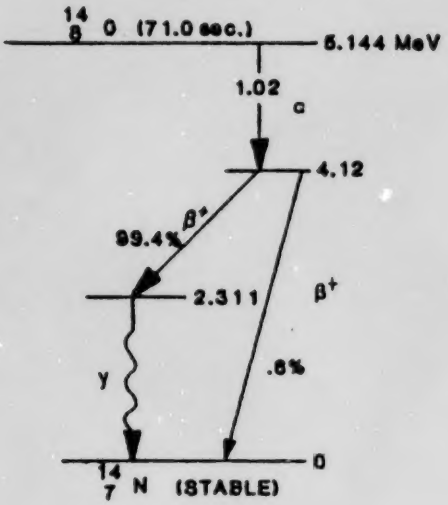


Figure 22-4-2.
Decay scheme for $^{14}_{8}O$.

In this decay scheme, a positron possessing an energy of 1.8 MeV (4.1 MeV - 2.3 MeV) and a gamma ray of 2.3 MeV are emitted.

Another reaction that can occur in an atom that has a proton excess is K-capture – the capture of an electron by the nucleus from the innermost atomic shell (the K-shell). The end effect for the nucleus is the same as for β^+ decay. The void created in the K-shell is filled by the capture of an outer orbital electron. Figure 22-4-3 depicts the typical K-capture process for $^{51}_{24}Cr$.

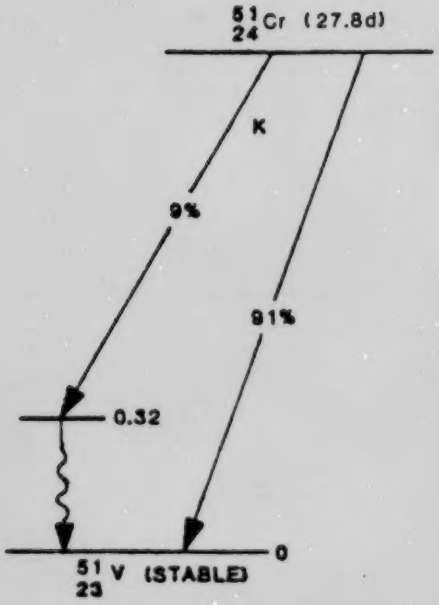


Figure 22-4-3.
Typical K-capture process.

The residual nucleus $^{51}_{23}V$ is left in an excited state, which can result from K-capture and particle emission. The energy difference between the excited nucleus and the residual nucleus (ground state) is, in most cases, immediately radiated in the form of one or more γ rays. For some nuclides, however, the excited intermediate nucleus lasts considerably longer in a quasi-stable state, decaying radioactively with a well-determined half-life. This quasi-stable state often is referred to as a "metastable" state.

ENERGY CHARACTERISTICS OF BETA DECAY

Nuclides that have the same mass number but different atomic numbers are called "isobars." ^{32}P and ^{32}S are isobars: their atomic numbers – and, hence, their energies – are fixed.

When ^{32}P undergoes β^- decay to ^{32}S as depicted in Figure 22-4-1, the difference in energy between them is a constant value. One could get the incorrect impression that beta decay energy, like energy emitted by alpha and gamma decay, is monoenergetic and that beta spectroscopy is a simple matter of measuring this monoenergetic radiation and using this value to identify the radionuclide. This, however, is not the case. This cannot be done because energy spectrum of β particles is continuous, not monoenergetic.

This characteristic of β-particle emission puzzled researchers until, in 1931, a scientist suggested that the β particle was not the only one emitted and that an undiscovered particle shared the total energy with the beta particle. This particle was named the "neutrino" (from the Italian "little neutron"). The neutrino carries no electric charge and has a mass approaching zero. The energy of the neutrino plus the energy of the beta particle equals the difference in energy between the two isobars, as indicated by Equation 22-4-4.

$$E_\beta + E_\nu = \Delta E \qquad \text{Eq. 22-4-4}$$

where

E_β = energy of the beta particle.
E_ν = energy of the neutrino.
ΔE = difference in energy (mass difference) between the two isobars.

The exact way in which the beta particle and the neutrino share energy is not fixed from one decay to the next. The beta particle may carry off all the energy or none of

it, resulting in a typical beta-energy distribution curve such as that in Figure 22-4-4.

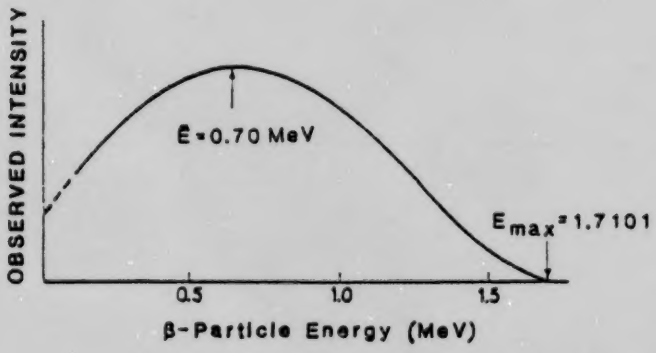


Figure 22-4-4. Simple β-particle energy-distribution curve for ^{32}P.

This figure represents the simple beta-particle energy distribution curve for ^{32}P. The energy of the beta particle is plotted as a frequency of beta particles having that energy. At the left-hand side of the figure, the beta particle is taking none of the energy, and the neutrino is taking almost all of it. At the extreme right-hand side of the figure, the beta particle is taking almost all the energy and the neutrino almost none. In a beta-particle energy distribution curve such as this, the curve can be extrapolated to the maximum energy that the beta particle reasonably could have. This energy is $E_{\beta max}$ and is equal to the energy difference between two adjacent isobars: $E_{\beta max}$ is the one unique feature of beta decay that can be identified and utilized to assist in the identification of a radionuclide. A very exact measurement of $E_{\beta max}$ is quite difficult, however, and involves an

extrapolation of a curve not exactly known. As a result of this uncertainty, the usefulness of this one unique characteristic of beta decay to identify radionuclides is rather limited. For these reasons, beta analysis is the least exact and least useful of the techniques employed in the identification of unknown radionuclides.

MEASURING $E_{\beta max}$

There are two methods by which $E_{\beta max}$ can be measured. The first method requires a high-vacuum system containing a very powerful magnet, with which the paths of beta particles are bent. The magnetic field divides the beta particles according to their kinetic energy. This instrument is referred to as a "magnetic spectrometer" (Figure 22-4-5). Within this instrument, a collimated beam of β particles is deflected through 180° by a strong magnetic field. The more energetic particles in the beam swing in a wider arc, and the less energetic ones travel over a shorter arc; therefore, the beam particles are spread out to form a spectrum deflected onto a strip of film.

These instruments are found primarily in research labs; they allow researchers to construct beta-particle spectra.

Another method, more practical and less expensive, is "Feather Analysis" – a comparison method for the routine determination of maximum beta range, in which the range of the unknown beta-emitting radionuclide is compared to the range of beta particles of a standard isotope. The range can be related to $E_{\beta max}$, the distinctive characteristic of the beta-emitting nuclide.

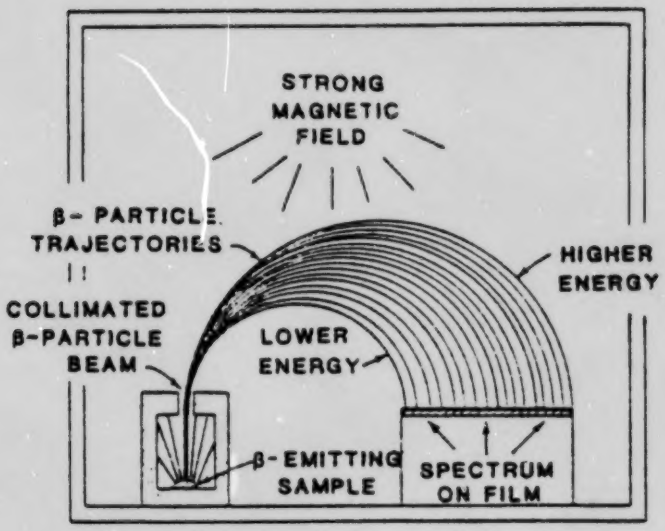


Figure 22-4-5. Simplified diagram of a magnetic spectrometer.

BETA ANALYSIS

Two methods can be employed. In one method, a beta-sensitive detector such as a GM or gas-flow proportional counter counts the activity of the unknown nuclide. Beta absorbers of varying thicknesses are placed between the nuclide and the window of the counter. Once the absorber thickness necessary to stop all the beta particles is known, the beta-particle range can be established. This range can be converted to $E_{\beta max}$ by use of a range energy curve of the type illustrated in Figure 22-4-6.

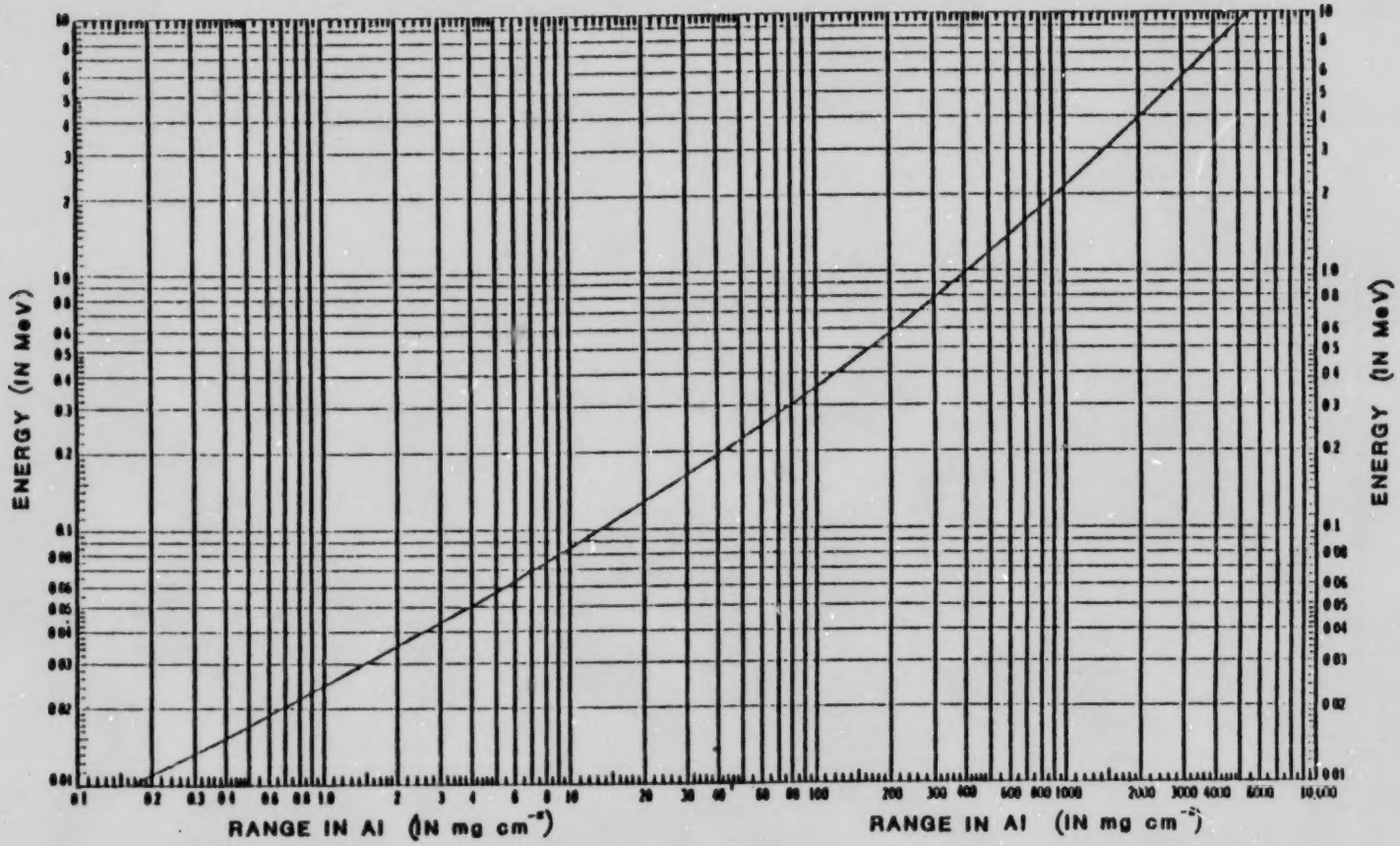


Figure 22-4-6. Range-energy relation for β particles and electrons in aluminum.

EXAMPLE

Given: An unknown beta-emitting nuclide that has a range of 476 mg/cm^2 in aluminum.

Find: Identity of the nuclide.

Solution: Refer to Figure 22-4-6. Energy for β particles having a range of 476 mg/cm^2 is 1.3 MeV. Use reference to find nuclide having this $E_{\beta max}$. This nuclide is Radium DEF.

Although this first method appears to be a fairly simple and straightforward one, it is impractical in many cases, unfortunately. For many nuclides, increasingly thicker absorbers are required, with a corresponding increase in statistical uncertainty and counting time.

The comparison method, with which the beta range of particles from a reference standard is compared to the range of those from an unknown nuclide, is a routine procedure for determination of beta-particle range. Radium DEF is a popular reference standard.

A sample of the radioactive unknown is prepared and mounted on a planchet and is counted as varying thicknesses of absorber are placed between the window of the counter and the nuclide. The sample is not moved as the activity is counted for each thickness of absorber. At least 5,000 total counts – preferably 10,000 – should be registered. The measurements are continued with increasingly thicker absorbers until there is no further appreciable decrease in activity. Data is entered into a table such as Table 22-4-1.

TABLE 22-4-1

Total Absorber Thickness	Total Counts	Activity cpm	Activity Corrected for Background
1.			
2.			
3.			
4.			
5.			

Total absorber thickness includes the absorption of the window of the counter, the absorption because of air between the sample and window, the thickness of the absorber, and the thickness of any cover placed over the sample for protection.

Background correction must include natural background radiation and the radiation caused by gamma emission and bremsstrahlung. The term "bremsstrahlung" is German and means, literally, "brake radiation," or the electromagnetic radiation emitted by an accelerated or decelerated charged particle. The activity corrected for background then is calculated and its value entered into the Data Table.

The same procedure is followed for the standard. The results for the standard are plotted on semilog graph paper. The corrected activity is plotted as the ordinate and the total absorber thickness (in mg/cm^2) as the abscissa. Figure 22-4-7 consists of a typical plot of absorption of RA-DEF β particles in matter. The linear portion of the curve is extrapolated to zero total absorber thickness.

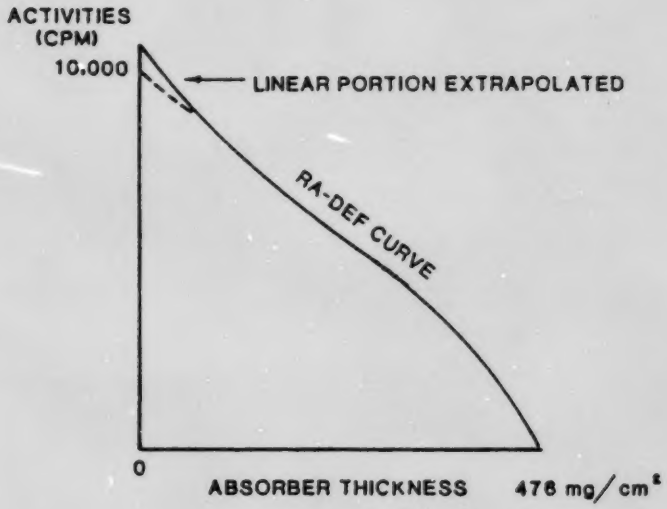


Figure 22-4-7.
Absorption curve for RA-DEF.

Data should be plotted as near to 476 mg/cm^2 (the range of RA-DEF in aluminum) as possible.

The abscissa then is divided into fractions of 1/10 of the range. Since the range is 476 mg/cm^2, the abscissa should be divided at intervals of 47.6 mg/cm^2 (Figure 22-4-8).

Lines are projected from each 0.1 interval of the range on the abscissa, up to the curve, and then to the ordinate. This procedure marks off activities on the ordinate as read from the curve. If activities were 10,000, 5,000, 2,500, 1,200, 600, 300, 150, 70, and 30 cpm, they would be marked as in Figure 22-4-9.

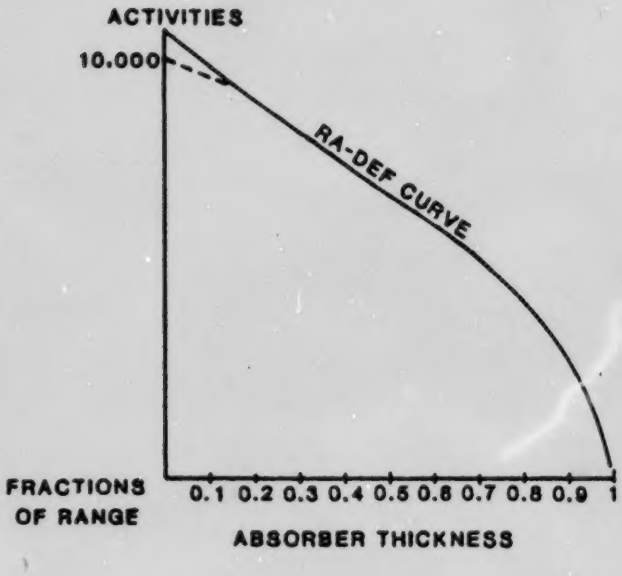


Figure 22-4-8.
Absorption curve for RA-DEF divided into 1/10 parts.

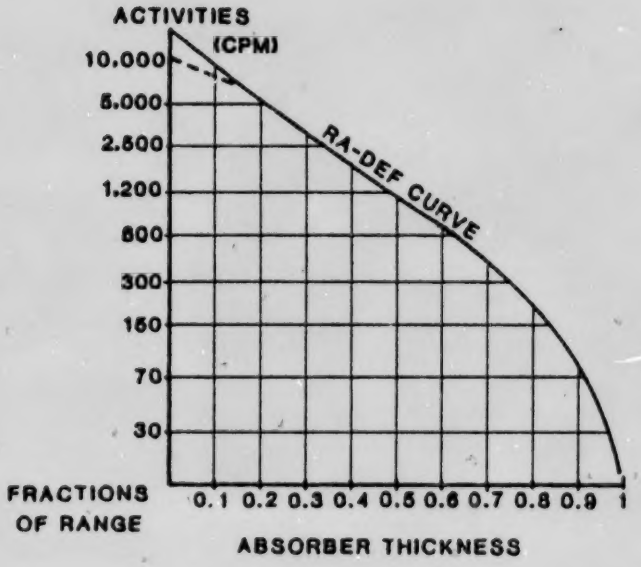


Figure 22-4-9. Activities marked off on ordinate.

These activities, when correlated with fractions of range in this way, allow the technician to calculate transmission factors for each 1/10 range. The technician assigns a transmission factor of 1 to the total activity (10,000 cpm in this case) and calculates a relative value for the other ordinate intercepts. The transmission factor is the fraction of the total number of beta particles penetrating the absorbers through 0.1, 0.2, 0.3, etc., of the total range (Figure 22-4-10).

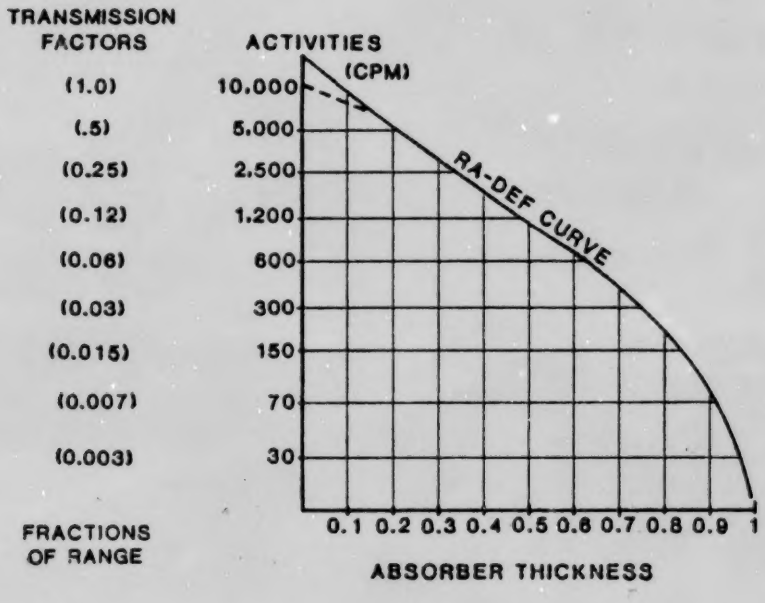


Figure 22-4-10. Transmission factors for RA-DEF.

The transmission factor is calculated by division of the activity at any point into maximum activity; for example $\frac{10,000}{10,000} = 1$, or again, $\frac{1,200}{10,000} = 0.12$, etc. A data table is constructed, and all data recorded in it (Table 22-4-2).

TABLE 22-4-2

Tenths of Range	RA-DEF Activity cpm	Transmission Factor
0	10,000	1.0
1	5,000	0.5
2	2,500	0.25
3	1,200	0.12
4	600	0.06
5	300	0.03
6	150	0.015
7	70	0.007
8	30	0.003
9	too low to record	---
10	0	0

The expected activity of the unknown nuclide at each 1/10th of its range is calculated by multiplication of the unknown activity at zero total absorber thickness by the transmission factors for aluminum just established, as in Equation 22-4-5:

Activity of unknown nuclide at each 1/10 range = Activity of nuclide at zero total absorber thickness x Transmission factors for each 1/10 range

Eq. 22-4-5

An additional section can be added to the data table depicted in Table 22-4-2. The activity of the unknown

nuclide at zero absorber thickness is entered in the first block. The activity of the unknown nuclide at each 1/10 of its range is calculated and entered in the appropriate block. If the activity of the unknown nuclide were 20,000 cpm, the range of activities would be entered as in Table 22-4-3.

TABLE 22-4-3

Tenths of Range	RA-DEF Activity (cpm)	Transmission Factor	Activity of Unknown Nuclide (cpm)
0	10,000	1.0	20,000
1	5,000	0.5	10,000
2	2,500	0.25	5,000
3	1,200	0.12	2,400
4	600	0.06	1,200
5	300	0.03	600
6	150	0.015	300
7	70	0.007	140
8	30	0.003	60
9	too low to record	--	--
10	0	0	--

Once the transmission factor is known, the activities of the unknown nuclide at each 1/10 of its range can be calculated by multiplication of the activity of the unknown nuclide at zero total absorber thickness by the transmission factors already established. The activities counted for the unknown nuclide then must be plotted against absorber thickness in the same manner as used for RA-DEF. (Refer to Figure 22-4-11.)

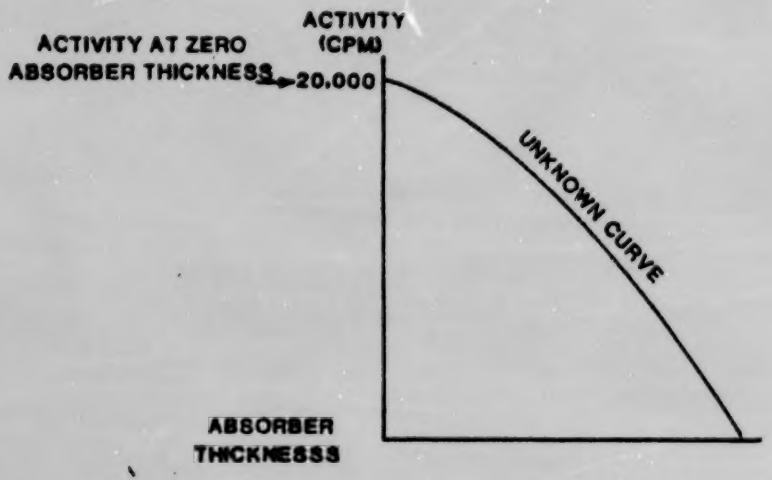


Figure 22-4-11. Absorption curve for unknown nuclide.

Points that correspond to these calculated activities are laid off along the ordinate of the unknown curve, as illustrated in Figure 22-4-12.

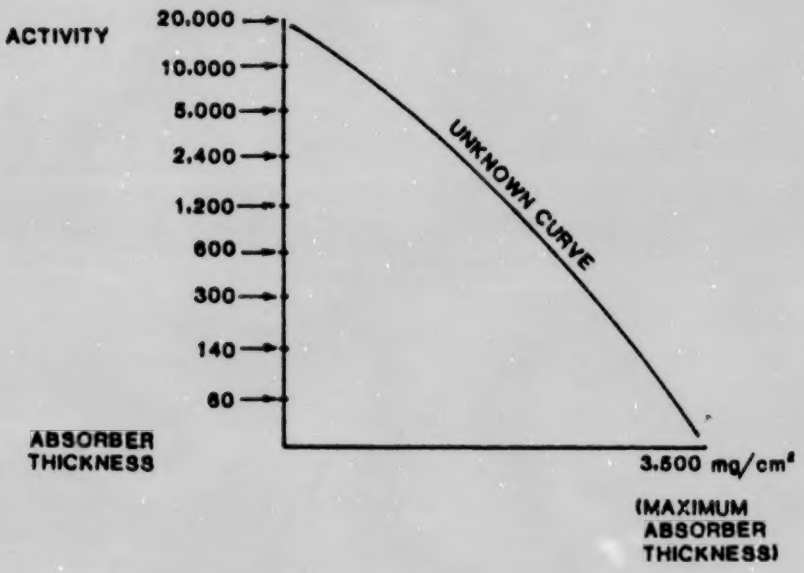


Figure 22-4-12. Maximum absorber thickness.

DETERMINATION OF ACTIVITIES

Lines are projected from these points to points on the curve and then to the abscissa. Points on the abscissa correspond to the absorber thicknesses, representing 0.1, 0.2, 0.3 of the range (Figure 22-4-13).

If absorbers having thicknesses of 3,500 mg/cm^2 to 100 mg/cm^2 were used, a data table such as the one in Table 22-4-4 could be constructed.

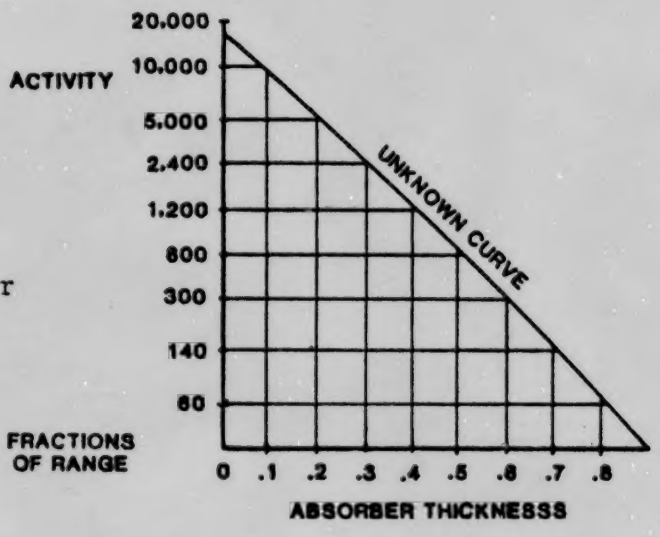


Figure 22-4-13.
Determining absorber thickness.

TABLE 22-4-4

Fraction of Range	Absorber Thickness mg/cm^2
0.1	100
0.2	300
0.3	650
0.4	1,000
0.5	1,350
0.6	1,700
0.7	2,000
0.8	2,350
0.9	2,700
1.0	3,500

The apparent range is calculated by dividing the absorber thickness by the fraction of the range, as indicated by Equation 22-4-6.

$$\text{Apparent Range} = \frac{\text{Absorber Thickness}}{\text{Fraction of the Range}} \qquad \text{Eq. 22-4-6}$$

Results for absorbers from 3,500 to 100 mg/cm^2 are given in Table 22-4-5.

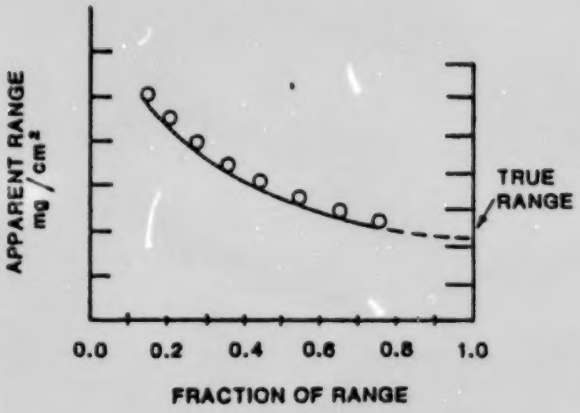
TABLE 22-4-5.

Fraction of Range	Absorber Thickness in mg/cm^2	Apparent Range in mg/cm^2
0.1	100	1,000
0.2	300	1,500
0.3	650	2,166
0.4	1,000	2,500
0.5	1,350	2,700
0.6	1,700	2,833
0.7	2,000	2,857
0.8	2,350	2,937
0.9	2,700	3,000
1.0	3,500	3,500

True range can be determined from a graph of the values for the apparent range versus fraction of the range and from

extrapolation of the curve to 10/10 of the range (Figure 22-4-14). This extrapolated value is the true range.

Figure 22-4-14.
Extrapolation to true range.



When the true range is known, the Glendenin equation, or a range-energy curve can be employed to determine $E_{\beta max}$.

The Glendenin equation (Equation 22-4-7) is utilized when energies are greater than 0.8 MeV and ranges are greater than 300 mg/cm².

$$E_m = \frac{R_m + 133}{542} \qquad \text{Eq. 22-4-7}$$

where

R_m = maximum range in mg/cm².
E_m = maximum energy of the beta particles in MeV.

EXAMPLE

Given: An unknown beta-emitting radionuclide exhibits a true range of 850 mg/cm^2.

Find: $E_{\beta max}$.

Solution: Use Equation 22-4-7.

$$E_m = \frac{R_m + 133}{542}$$

$$= \frac{850 + 133}{542}$$

$$= \frac{983}{542}$$

$$E_m = 1.81 \text{ MeV}$$

$$E_{\beta max} = 1.81 \text{ MeV}.$$

A range-energy graph such as the one in Figure 22-4-6 can be drawn in order to relate those ranges and energies too low in value to be calculated from Equation 22-4-7. A table of isotopes allows the identification of the nuclide by its $E_{\beta max}$ value.

EXERCISES

1. From Figure 22-4-6, find $E_{\beta max}$ for the following ranges in Al:
   - 8 mg/cm^{-2}
   - 320 mg/cm^{-2}
   - 260 mg/cm^{-2}
2. Refer to a table of isotopes, and diagram the expected beta spectrum for the following nuclides. (A table of

isotopes is available in such reference sources as those by Lederer and Weast, listed in the "Reference Materials" of this module.)

^{32}P

^{33}S

^{3}H

^{90}Sr

3. Calculate $E_{\beta max}$ for a nuclide having a range of 500 mg/cm^2.
4. Calculate the expected maximum range from the decay of ^{35}S.

MATERIALS

GM or Gas Flow Proportional counter with scaler.
Unknown beta-emitter.
RA-DEF source.
Calibrated aluminum absorbers.
Sample holder.
Semilogarithmic graph paper.
Table of Isotopes (available in such reference sources as those by Lederer and Weast, listed in the "Reference Materials" section of this module).

FEATHER ANALYSIS

1. Prepare a sample of Radium DEF, place in holder, and adjust geometry to achieve a count of 20,000 cpm.
2. Measure activity of the sample for each absorber in order that total count is at least 10,000 cpm. Enter results in Data Table 22-4-1. Total absorber thickness includes the absorption of the counter window, the absorption because of air, and the thickness of any protective cover. Continue counting until net rate falls below 100 cpm net.
3. Take background reading, and enter corrected activities in Data Table 22-4-1.
4. Plot the results of the RA-DEF standard on semilog graph paper. Corrected activity is plotted as the ordinate and the total absorber thickness as the abscissa.
5. Extrapolate this curve to zero total absorber thickness and as near 476 mg/cm^2 as possible (range of RA-DEF in aluminum).
6. Divide the abscissa into fractions of 0.1 of the range.
7. Project lines from each interval of 0.1 of the range on the abscissa up the curve and then to the ordinate. Mark off activities on ordinate as read from the curve.
8. Assign a transmission factor of unity to the total activity, and then assign a relative value to each of the other ordinate intercepts by dividing the activity into the maximum activity. Enter data in Data Table 22-4-2.
9. From Equation 22-4-5, calculate the activity of the unknown nuclide at each 0.1 of its range. Complete Data Table 22-4-2.
10. Plot activities for unknown nuclide against absorber thickness in the same manner as used for RA-DEF.

11. Lay off points corresponding to the calculated activities of the unknown nuclide on ordinate of curve plotted in Step 10.
12. Project lines from these points to points on the curve and then to the abscissa. These points on the abscissa correspond to absorber thicknesses representing 0.1, 0.2, 0.3, etc., of the range. Entere data into Data Table 22-4-3.
13. Calculate apparent range from Equation 22-4-6. Complete Data Table 22-4-3.
14. Plot values of apparent range against fraction of the range, and extrapolate the curve to 10/10 of the range. The extrapolated value is the true range.
15. Use true range to determine $E_{\beta max}$ from either Equation 22-4-7 or from range-energy curve in Figure 22-4-6.
16. From value of $E_{\beta max}$, use Table of Isotopes to identify the nuclide.

DATA TABLES

DATA TABLE 22-4-1

	Total Absorber Thickness	Total Counts	Activity cpm	Activity Corrected for Background
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				

DATA TABLE 22-4-2

Tenths of Range	RA-DEF Activity (cpm)	Transmission Factor	Activity of Unknown Nuclide (cpm)
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			

DATA TABLE 22-4-3

Fraction of Range	Absorber Thickness in mg/cm^2	Apparent Range
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		

Chase, Grafton D. and Rabinowitz, Joseph L. Principles of Radioisotope Methodology. 3rd ed. Minneapolis: Burgess Publishing Company, 1967.

Friedlander, G.; Kennedy, J.W.; and Miller, J. Nuclear and Radiochemistry. New York: John Wiley and Sons, 1964.

Lederer, C.M.; Hollander, J.M.; and Perlman, I. Table of Isotopes. 6th ed. New York: J. Wiley and Sons, 1968.

Wang, C.H.; Willis, D.L.; and Loveland, W.D. Radiotracer Methodology in the Biological, Environmental and Physical Sciences. Englewood Cliffs, NJ: Prentice-Hall, 1975.

Weast, Robert C., ed. CRC Handbook of Chemistry and Physics. 55th ed. Cleveland, OH: CRC Press, 1974.

Section I: Matching.

Match the term in Column A with the description in Column B by placing the appropriate letter in the parenthesis:

"A"		"B"	
1.	Positron (_____)	A.	Compares the range of the beta particles of an unknown nuclide to the range of those of a standard nuclide.
2.	Negatron (_____)	B.	Deflects a collimated beam of β-particles through 180°.
3.	β^- emission (_____)	C.	The unique characteristic of beta decay.
4.	β^+ emission (_____)	D.	Carries no electric charge and has a mass approaching zero.
5.	K-capture (_____)	E.	Nuclides having same mass number but different atomic numbers.
6.	Isobar (_____)	F.	An electron from the innermost shell is drawn off by the nucleus.
7.	Neutrino (_____)	G.	The residual nucleus moves to a low atomic-number element, and the atomic number is reduced by one.
8.	$E_{\beta max}$ (_____)	H.	Increases the atomic number of the residual nucleus.
9.	Magnetic spectrometer (_____)	I.	The term "beta particle" commonly used for this particle.
10.	Feather Analysis (_____)	J.	Carries a positive charge.

Section II: The Writing of Equations.

11. Write a sample decay scheme for the following:

    a. β^+ emission

    b. β^- emission

    c. K-capture

12. Write an equation that defines –

    $E_{\beta max}$

Section III: Use of the Energy Distribution Curve.

From an energy distribution curve (Figure 22-4-5), determine $E_{\beta max}$ for nuclides having the following ranges in aluminum:

13. 0.2 mg/cm^{-2} _____

14. 6 mg/cm^{-2} _____

15. 400 mg/cm^{-2} _____

Section IV: Descriptive Problems.

16. Explain why beta analysis is the least exact and least useful of the techniques for nuclide identification.

17. Explain when beta analysis should be utilized.

18. Describe how the magnetic spectrometer sorts beta particles by energy.

19. How is an unknown nuclide identified by Feather Analysis?

Section V: Calculations.

20. Given: Transmission factors of 1.0, 0.5, 0.25, 0.12, and 0.06; an unknown nuclide with an activity of 13,000 at zero total absorber thickness.

Find: The apparent range of the unknown nuclide at each 0.1 range.

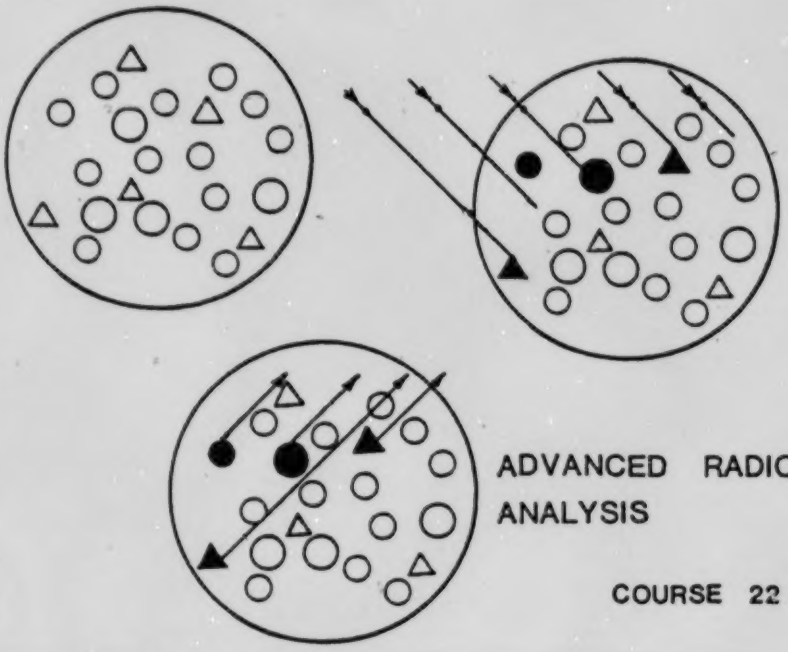
21. Given: An unknown beta-emitting nuclide having a true range of 700 mg/cm^2.

Find: $E_{\beta max}$.

NUCLEAR TECHNOLOGY

MODULE 22 - 5

RADIOCHEMICAL METHODS OF ANALYSIS BY
LIQUID SCINTILLATION COUNTING



ADVANCED RADIONUCLIDE
ANALYSIS

COURSE 22

CORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 22 ADVANCED RADIONUCLIDE ANALYSIS

MODULE 22-1	Survey of Methods Used in Radionuclide Analysis (Sample Collection & Analysis)
MODULE 22-2	Gamma Spectrometry
MODULE 22-3	Alpha Spectrometry
MODULE 22-4	Beta Spectrometry
MODULE 22-5	Radiochemical Methods of Analysis by Liquid Scintillation Counting
MODULE 22-6	Neutron Activation Analysis
MODULE 22-7	Determination of Specific Nuclides (In the Environment)

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INTRODUCTION

Liquid Scintillation Counting (LSC) is the determination of nuclear disintegration rate by the measurement of light produced by the conversion of disintegration energy into light. The liquid scintillation counter is a device in which light flashes produced by a liquid scintillator when exposed to ionizing radiation are converted by a photomultiplier into electrical pulses that enable the number of ionizing events to be counted. The amount of light in each pulse determined with the photomultiplier tube represents a measure of the energy deposited in the scintillator. The ability to evaluate these energies provides a means of sorting radiations from different sources and of identifying and determining the magnitudes of the radiation sources.

LSC often is employed in the detection and identification of radionuclides. This method is especially useful and sensitive in the detection of low-energy beta emitters such as tritium, carbon-14, and sulphur-35.

This module discusses LSC and its use in the detection of weak beta-emitting radionuclides.

MODULE PREREQUISITES

The student should have completed a course in basic algebra and Course 11, "Radiation Detection and Measurement."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Define the following terms:
   - Luminescence.
   - Fluorescence.
   - Phosphorescence.
   - Scintillator.
   - Inorganic crystals.
   - Organic scintillators.
   - Liquid scintillators.
   - Liquid scintillation medium.
   - Plastic scintillator.
   - Photomultiplier tube.
   - Photocathode.
   - Dynode.
   - Quenching.
   - Chemical quenching.
   - Dilution quenching.
   - Self quenching.
   - Color quenching.
   - Optical quenching.
   - Light guide.
   - Fluor.
2. List the four types of scintillators, and describe their distinguishing characteristics.
3. Describe typical liquid scintillation mediums. List the primary and secondary solvents and solutes.
4. Sketch a photomultiplier tube, label its components, and describe its operation. Include the following:
   - A description of the flow of electrons through the tube.

- A description of the typical gain of a tube and of the relationship of the gain to the number of electrons emitted by the photocathode and the quantity of light incident on the photocathode.

5. Describe the interaction of energetic electrons when a beta-emitting radionuclide is placed in the liquid scintillation medium.
6. Describe chemical, dilution, self, optical, and color quenching and the effect of quenching on counting efficiency.
7. Define coincident light and explain its use in the reduction of background radiation.
8. Describe the effect of different materials for sample vials on background radiation.
9. Draw and label a block diagram of a typical liquid scintillation counter, and describe its operation.
10. List five characteristics of a primary solvent.
11. Describe uses of the secondary solvent and the primary and secondary solute.
12. List and describe two methods for the determination of counting efficiency.

SCINTILLATION DETECTORS

Scintillation detectors make use of the physical property termed "luminescence" – the absorption of energy (in this case, from nuclear radiations) by a substance, resulting in re-emission of that energy in the form of visible, or near visible, light. In most cases, this light is emitted almost instantaneously after absorption of the radiation, but in some cases emission may be delayed. If emission of light occurs within 10^{-8} seconds of the time of excitation, the process is called "luminescence." If emission of light lasts longer than, or takes place after, 10^{-8} seconds, the process is called "phosphorescence." Phosphorescent materials are seldom utilized in nuclear counting work. Luminescent materials, called scintillators, often are coupled with various devices, such as photomultiplier tubes, to make possible the detection of low-level energies.

The four types of scintillators are –

- Inorganic crystals.
- Organic crystals.
- Liquid scintillators.
- Solid scintillators (plastic).

The common scintillators, their properties, and their uses are given in Table 22-5-1.

TABLE 22-5-1.
PROPERTIES OF COMMON SCINTILLATORS
(Activators in Parentheses)

Material	Type	Main Use	Density g/cm³	Wavelength of Light Emitted, Nanometers	Duration of Light Emission, μsec	Relative Pulse Height
NaI(Tl)	Inorganic Crystal	Gamma Counting	3.7	410	0.25	2.1
KI(Tl)	"	"	3.2	400	5.0	0.8
Cs(Tl)	"	"	4.5	White	1.0	0.5
ZnS(Ag)	"	Alpha Counting	4.1	450	10.0	2.0
LiI(Eu)	"	Neutron Detection	4.1	440	1.0	0.75
Anthracene	Organic Crystal	Beta Counting	1.3	440	0.03	1.0
Stilbene	"	"	1.2	410	0.008	0.6
Plastic	Solid	"	1.1	350-450	0.005	0.35
p-Terphenyl	Liquid	"	0.9	350-450	0.003	0.35

TYPES OF SCINTILLATORS

The first five materials listed in Table 22-5-1 are inorganic crystals, which are inorganic salts (primary alkali halides) that form regular crystal lattices. If the lattices have certain imperfections, such as physical defects or deliberately-introduced impurities, the crystal will scintillate when exposed to radiation. These are the most common of all scintillators and are in widespread use in gamma detectors. The most common inorganic crystal for scintillation counting is sodium iodide.

Organic scintillators are employed primarily in beta counting. They generally are aromatic hydrocarbons that contain benzene rings. The most common organic crystal is

anthracene, a coal-tar derivative. Both organic and inorganic crystals have a limited usefulness for the detection of weak beta particles because the particles are absorbed by the container that holds the crystal before they can reach the scintillator.

Liquid scintillators are not limited by this problem because the source can be placed directly in them, thereby increasing sensitivity. These scintillators usually consist of a solution of an organic scintillator material in an organic solvent. The active portion is an aromatic hydrocarbon similar to those of organic crystal scintillators.

The great advantage of liquid scintillators is that they can be mixed with water or other solutions and used to count low-energy beta particles from such weak beta emitters as ^{14}C (0.156 MeV_{max}) and ^{3}H (0.0186 MeV_{max}). The counting of such low-energy radiations would be impossible otherwise. The maximum range of a tritium beta particle in water is, for example, only about 0.25 mm; therefore, unless tritium is in intimate contact with the scintillator, no interaction will occur. Actual immersion of the radionuclide in the liquid scintillant solution provides this intimate contact.

There are many liquid scintillation media, a common one being 5 grams of p-terphenyl, dissolved in one liter of solvent (dioxane, in many cases). The p-terphenyl is the fluorescing substance and is known as the "primary solute." It is dissolved in a much larger quantity of organic solvent (dioxane). The radioisotope sample either is dissolved or is suspended in the solution. Other liquid scintillation media include PPO (2,5-diphenyl-oxagole) and BBOT 2,5-bis-2-(5-t-butyl-benzoxagolyl)-thiophene, which usually are dissolved in xylene, toluene dioxane, or other

aromatic solvents. PPO, BBOT, and p-terphenyl are compounds that contain six carbon benzene (phenyl) rings.

The final category of scintillators consists essentially of plastic organic solutions called "solid scintillators." They sometimes are employed in beta counting, but they have much lower efficiency than organic crystals of the same size.

SCINTILLATION DETECTOR COMPONENTS

Scintillators are utilized in conjunction with electronic devices known as "photomultipliers" (Figure 22-5-1).

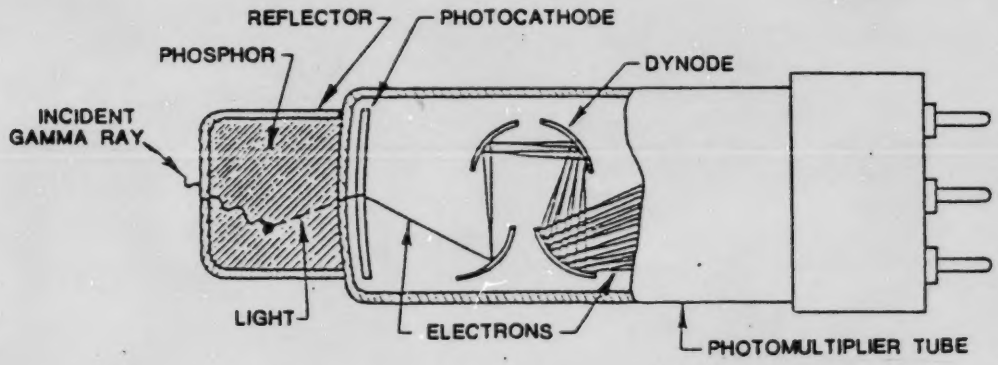

Figure 22-5-1. Scintillation phosphor and photomultiplier tube.

A photomultiplier tube is a combination of an ordinary photoelectric tube and an electron multiplying device. Light from a scintillator is transmitted, usually through a light pipe, to the face (photocathode) of the tube. This photocathode is constructed of a metal or metallic compound that emits electrons when struck by light. Electrons emitted by the photocathode are attracted to the first of a series of electrodes, whose primary function is the secondary emission of electrons. These electrodes are referred to as dynodes.

Dynodes are maintained at a positive potential in respect to the photocathode. Each dynode emits electrons when struck by an original electron and acts as an electron multiplier. A gradient of approximately 100 volts is maintained between each successive pair of dynodes, and the dynodes are shaped so that electrons emitted from one are focused onto the next.

The typical multiplication, or gain, of a photomultiplier tube is about 10^6; electrons emitted by a photocathode are amplified about a million times and then collected at the anode. The number of electrons collected is directly proportional to the number of electrons emitted by the photocathode. The number emitted by the photocathode is directly proportional to the amount of light that falls on it; and, of course, the amount of light created is directly proportional to the energy of the original radiation.

Electronic pulses are fed through an amplifier to single-channel analyzers. The electronics of this portion of the system are essentially the same as those described for gamma-ray analysis in Module 22-2.

The specifics of this system are discussed later in this module.

LIQUID SCINTILLATION COUNTING (LSC)

LSC involves the interaction of energetic electrons with a solute and solvent system. The primary solute is a fluorescent substance (sometimes referred to as the "primary scintillant") dissolved in a larger quantity of organic solvent. Fluorescence is the property of many substances of absorbing light of one wavelength and in its place emitting light of another wavelength or color. When radiation

interacts with these substances, small flashes of visible light (a scintillation) are produced. This process is the basis for the operation of scintillation detectors.

Figure 22-5-2 depicts the interaction of the energetic electrons when a beta-emitting radionuclide is placed in the solution.

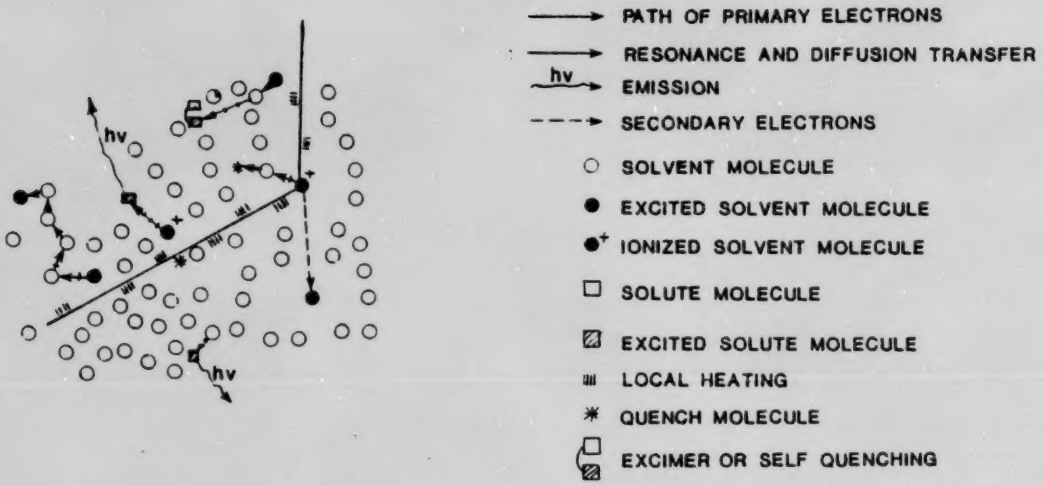

Figure 22-5-2. Energetic electron excitation processes of a two-component scintillator solution (solvent and solute).

As the beta particle travels through the solvent, it excites the solvent, either by ionization or by collisional excitation. This process requires only about 10^{-10} seconds. The solvent remains excited for a longer time and transfers its energy to the primary scintillant. The excited solute molecules return to the ground state by emitting photons that have a wavelength in the visible or ultraviolet region.

Because the emission peaks of the primary scintillant may not match the wavelengths to which the photocathodes are sensitive, a secondary scintillant often is included. Figure 22-5-3 illustrates the relationship between photocathode

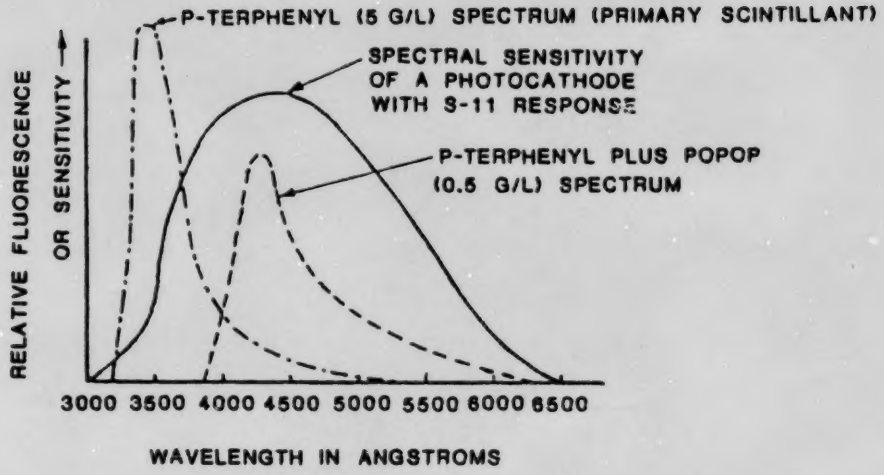

Figure 22-5-3. Fluorescence spectra of primary and secondary solutes (in toluene solvent) and photocathode sensitivity.

sensitivity and the spectra of primary and secondary solutes in toluene solvent. When a secondary scintillant such as POPOP is included, the spectrum more closely matches the spectral sensitivity of the photocathode. POPOP is p-bis [2(5-phenyloxazolyl)]Benzene. Some phototubes may not require a secondary scintillant.

A certain reduction of the efficiency of the energy transfer process, called "quenching," can occur. There are two types of quenching – chemical and self quenching. Several other phenomena have the same result (the reduction of the number of light quanta seen by the photomultiplier tube). These latter phenomena sometimes are referred to as "dilution," "optical," or "color" quenching. Quenching results in decreased light and, consequently, in a decreased output pulse in the photomultiplier. The extent of quenching varies with different sample materials.

Chemical quenching occurs when the molecules of components in the scintillation solution interact with the

molecules of the fluorescing substance and absorb their energy before it can be emitted as light. Dilution quenching is caused by the dilution of the fluorescing substance by the sample. This process reduces the number of beta-particle/solvent-molecule interactions and, hence, the probability of scintillation events. Self quenching occurs when the concentration of solute molecules is too high, causing the molecules of certain types of solutes to interact with other solute molecules and dissipate their energy as heat – not as light. In color quenching, colored sample material absorbs light and decreases the output of light per beta particle. Optical quenching occurs when the outside of the sample vial fogs, reducing the counting efficiency of the sample.

"Quenchers" are substances that possess a strong quenching action. They frequently have compounds that contain oxygen; for example, water, alcohols, betones, ethers, and halogen species such as chlorine or bromine. There have been partially-successful attempts to remove them from solutions chemically. A more practical approach is the determination of the extent of quenching and the adjustments for it. This approach is discussed later in this module under the heading "Determination of Counting Efficiency."

INSTRUMENT ASSEMBLIES FOR LIQUID SCINTILLATION COUNTING

Figure 22-5-4 illustrates a detector assembly in a typical liquid scintillation counter. Two photomultipliers are mounted in such a manner that they are sealed off from external light and that their open ends face the sample vial. Two photomultipliers are required, since one beta-particle/solvent-molecule interaction will result in more than one photon of light.

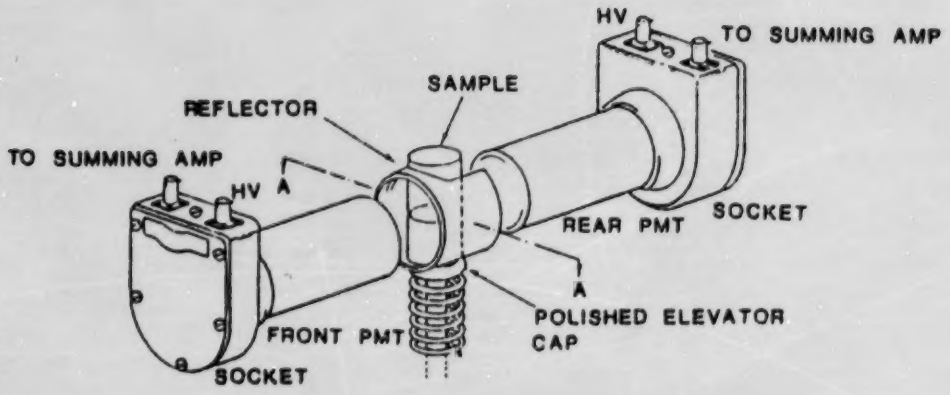

Figure 22-5-4. Components of a liquid scintillation counter.

The photomultiplier tubes are coupled to the sample vial by a plastic sleeve, or "light guide." The inside surface of the light guide is coated with a material that reflects light; such a material may consist of vacuum-deposited aluminum, titanium oxide, or magnesium oxide (Figure 22-5-5).

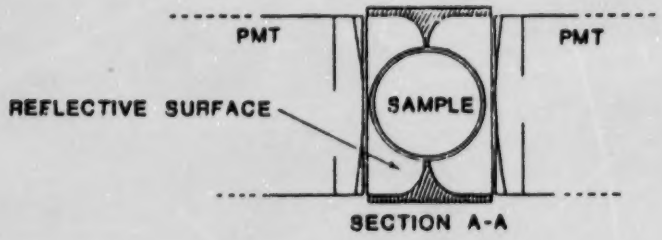

Figure 22-5-5. Detector assembly in a typical liquid scintillation counter. Courtesy of Beckman Instruments, Inc.

Light must enter both photomultiplier tubes at the same time to be called a "count." This effect, termed coincidence, lowers background radiation because most background radiation in a photomultiplier tube comes from background

noise that occurs in one tube at a time, but never in both at the same time.

The sample vial itself is normally constructed of a special low-potassium glass or Teflon to decrease the background count rate, since ordinary glass contains quantities of the radioisotope ^{40}K.

Figure 22-5-6 is a simplified block diagram of a liquid scintillation counter. Two photomultipliers, supplied with high voltage, face the sample vials from opposite sides. Coincident light creates pulses in each tube. Thermal emission of light from the photocathode, a normal phenomenon, would cause false counts were it not for the coincidence requirement. This problem is reduced by two photomultiplier tubes that feed to the coincidence logic box.

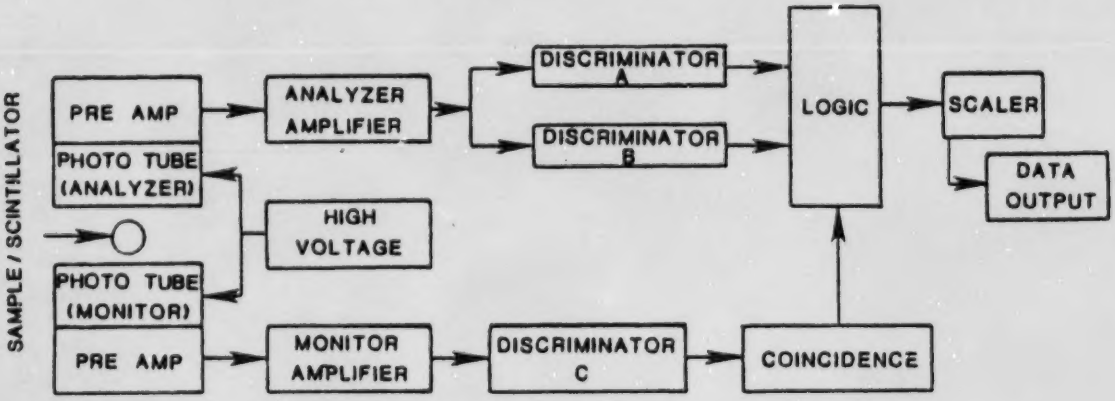

Figure 22-5-6. Simplified block diagram of a single-channel liquid scintillation counting system that employs coincidence logic.

The scintillations produced by the two betas are seen by the phototubes and simultaneously appear at the inputs to the monitor and analyzer amplifiers. If sufficiently large, pulses from the monitor amplifier pass discriminator C and go into the coincidence logic circuit. Analyzer amplifier

pulses that exceed the setting of discriminator A, but not B, pass through the logic and are counted. Pulses that exceed the level of B are killed in the logic circuit. Only pulses that occur simultaneously in both photomultiplier tubes are counted. Very large pulses – as from cosmic rays – are not counted because they have been killed in the logic circuit; thermal noise pulses are not counted because they seldom occur simultaneously in both multipliers.

SOLVENTS AND SOLUTES FOR LIQUID SCINTILLATION COUNTING

The primary solvent absorbs energy from beta particles and transfers the energy to the fluorescing solute (sometimes called the "fluor"). A primary solvent must exhibit the following characteristics:

- Have good energy-transfer characteristics.
- Absorb a minimum amount of the light emitted from the fluor.
- Be a good chemical solvent.
- Be free of contaminants that may reduce detector efficiency through a quenching effect.

Toluene, xylene, and dioxane have been mentioned as efficient primary solvents.

Sometimes, a secondary solvent must be used because some radioactive samples are not soluble in the primary solvent. The addition of ethanol, for example, improves the ability of toluene to mix with samples that contain water. Secondary solvents also can bind inorganic substances in order to make them soluble in the primary solvent.

The primary solute, or "fluor," converts excitation energy into light. PPO, BBOT, and p-terphenyl are common primary solutes.

The secondary solute shifts the wavelength of light emitted by the primary solute to a value at which the photocathodes are sensitive. Only very small amounts of secondary solute are required. POPOP is the most popular secondary solute.

DETERMINATION OF COUNTING EFFICIENCY

Because of quenching, a determination of counting efficiency for every prepared sample is required. Several methods are available.

THE INTERNAL STANDARD METHOD

One of the simplest, but most time-consuming, methods for determining the extent of quenching involves the use of an internal standard. The standard may be any calibrated solution of the nuclide being assayed. After measurement of the unknown sample activity R_u, an aliquot of standard solution is added to the sample; and the activity is measured again. The result is the total activity $R_t = R_u + R_s$. Since the absolute decay rate A_s of the standard is known, the absolute sample activity A_u is given by Equation 22-5-1.

$$A_u = A_s \frac{R_u}{R_s} = \frac{A_s R_u}{R_t - R_u} \qquad \text{Equation 22-5-1 (Equation = Eq.)}$$

For improved accuracy, quenching caused by the added reference standard also must be taken into account. After R_u and R_t are measured as described above, an amount of non-radioactive standard, chemically equivalent to the radioactive standard, is added; and the decrease in count rate R_t is noted. The process may be repeated if necessary to provide ample data with which to extrapolate to R_t^o, the theoretical activity of the sample plus a massless standard.

THE EXTERNAL STANDARD METHOD

The external standard method was developed to provide a faster determination than possible with the internal method.

The external standard method consists of a gamma-ray emitter (the standard) stored in a shielded chamber in the liquid scintillation counter. The gamma-ray source irradiates the sample. Although the external source provides only a relative correction or normalization of the data (rather than a value of absolute decay rate as with the internal standard), it is fast and precise. When absolute decay rates of the sample are required, the external source can be calibrated against an internal standard for the particular scintillation system used.

Counting efficiency of a prepared sample can be calculated for the external standard method by use of Equation 22-5-2:

$$\varepsilon = \frac{\text{cpm}}{\text{dpm}} \qquad \text{Eq. 22-5-2}$$

where:

ε = counting efficiency of sample.

cpm = counts per minute of quenched standard.

dpm = disintegrations per minute (activity; decay rate in counts) of quenched standard.

This equation will be important in the "Procedures" section of this module.

Since the energy spectrum of the Compton-derived electrons resembles the energy spectrum of weak beta emitters, the counting efficiency of a weak beta emitter can be correlated with the counting rate of a given gamma source. A standard correlation curve then can be prepared for the calibration of counting efficiency. Such a curve consists of a plot of the net cpm from the external standard against the counting efficiency of the quenched standard, as in Figure 22-5-7.

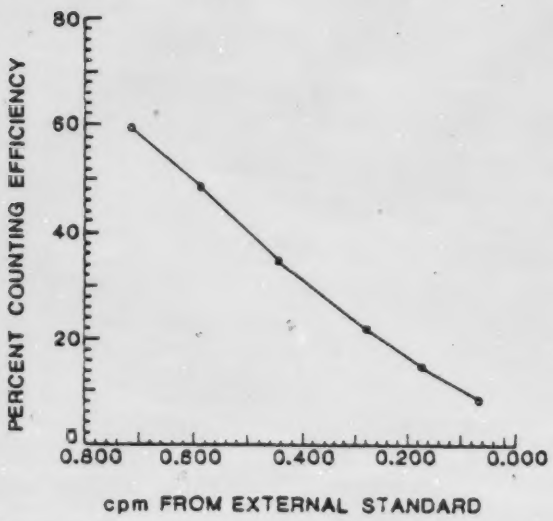

Figure 22-5-7. Quench correction curve for external standard method.

THE PULSE HEIGHT SHIFT METHOD

The pulse height shift method is a stastical evaluation of counting performance. The method involves the sorting of pulses produced by a detector according to size; only those pulses of the size that would be produced by the sample are accepted. A relationship for the evaluation of counting procedures and equipment to determine the optimum conditions of counting efficiency and background count is given by the ratio in Equation 22-5-3.

$$\frac{R_s^2}{R_b} \qquad \text{Eq. 22-5-3}$$

where:

R_b = background counting rate.

$R_s = R_t - R_b$ = net sample counting rate.

R_t = total counting rate (sample plus background).

This equation expresses the ratio of the square of the sample activity to the background activity. The greater the value of the ratio, the better the counting conditions.

Equation 22-5-3 can be simplified for use with a two-channel LSC as indicated by Equation 22-5-4, which is similar to Equation 22-5-2.

$$\varepsilon = \frac{cpm}{dpm} \qquad \text{Eq. 22-5-4}$$

where:

ε = counting efficiency of sample used in pulse height method.

cpm = counts per minute of sample minus counts per minute of quenched standard.

dpm = disintegrations per minute (activity; decay rate in counts) of quenched standard.

This method takes advantage of the fact that when quenching occurs, the average pulse height of an energy spectrum decreases. A standard quench correction curve can be constructed from data gathered in the following steps: (1) A dual liquid scintillation spectrometer is set up in such a manner that the discriminators obtain a defined ratio between the sample counting rate (in one channel) minus the background counting rate in the other channel. (2) The counting rate (in dpm) for varying amounts of quenching agent is determined. (3) A standard quench correction curve is constructed. A typical curve is illustrated in Figure 22-5-8.

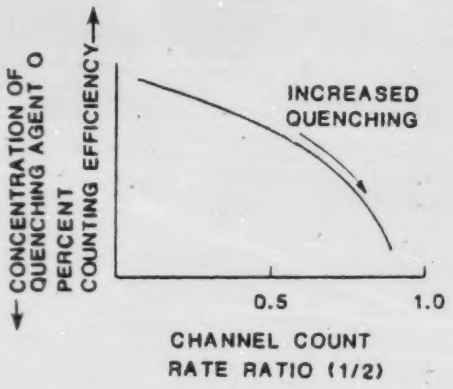

Figure 22-5-8. Quench correction curve for the pulse height shift method.

A quench correction curve for each of the weak beta emitters ^{14}C and ^{3}H also covers many of the other common chemical quenching agents.

EXERCISES

1. List two reasons why LSC is used in place of gas-filled or proportional counters.
2. What is the efficiency of a 100,000-dpm sample that counts 38,500 counts in 0.5 minute?
3. How does the pulse height shift method determine the counting efficiency of a sample?

MATERIALS

1 solution, consisting of 1 liter of scintillation grade toluene, 10 grams of PPO, and 0.1 gram of POPOP.

1 set of ^{14}C or ^{3}H quenched standards.

1 LSC counter (such as a Beckman LS-255 or Nuclear Chicago Mark II).

LSC vials with tops. Vials should be made of low-potassium glass.

PROCEDURES

1. Make a series of at least 10 samples by diluting a ^{14}C or ^{3}H (quenched standards) solution with the LSC solution. Fill vials to about 75% full. Do not add radionuclide to one vial. Use this vial to determine background.

2. Set up LSC to count either ^{3}H or ^{14}C. Place an appropriate set of these quenched standards in the counter.
3. Acquire data for quench curve.
   a. If counter has external standard, use the count rate data from quenched standards to complete Data Table 22-5-1. Plot net cpm from the external standard against the counting efficiencies of the quenched standard.
   b. If pulse height shift method is used, complete Data Table 22-5-2; and plot the ratios of the net counts in channel 1 to channel 2 against the counting efficiencies of the quenched standard.
4. Count samples, following procedures suggested by instructor. Record data in Data Table 22-5-3.

DATA TABLE 22-5-1

QUENCHED CURVE DATA
(LSC with External Standard)

Quenched Standard #	cpm of Quenched Standard (1)	dpm of Quenched Standard (2)	$\varepsilon = \frac{\text{cpm (1)}}{\text{dpm (2)}}$	Count Rate of External Standard

DATA TABLE 22-5-2

QUENCH CURVE DATA FOR USE WITH
PULSE HEIGHT SHIFT METHOD

Quenched Standard #	cpm Channel 1	cpm Channel 2	dpm of Quenched Standard (3)	$\varepsilon = \frac{\text{cpm (2)}}{\text{dpm (3)}}$	Ratio $\frac{1}{2}$

DATA TABLE 22-5-3

NET COUNT DATA

Sample #	Gross dpm	– Background dpm	= Net dpm

Chase, Grafton D. and Rabinowitz, Joseph L. Principles of Radioisotope Methodology. 3rd ed. Minneapolis: Burgess Publishing Company, 1967.

Horricks, D. L. "Liquid Scintillation Counting." In Survey of Progress in Chemistry, Vol. 5, A. F. Scott, ed. New York: Academic Press, 1969.

Morgan, K. Z. and Turner, J. E., ed. Principles of Radiation Protection: A Textbook of Health Physics. 2nd ed. Huntington, NY: Robert E. Krieger Publishing Company, 1973.

Shapiro, Jacob. Radiation Protection: A Guide for Scientists and Physicians. Cambridge, MA: Harvard University Press, 1972.

Wang, C. H.; Willis, D. L.; and Loveland, W. D. Radiotracer Methodology in the Biological Environmental and Physical Sciences. New Jersey: Prentice-Hall, 1975.

A. Matching.

Match the word in column A with the appropriate phrase in column B by placing the Arabic numeral from column A into the matching parenthesis in column B.

COLUMN A	COLUMN B
1. Luminescence	a. ( ) the fluorescing solute.
2. Phosphorescence	b. ( ) couples photomultiplier to sample vial.
3. Scintillator	c. ( ) counting efficiency reduced when sample vial fogs.
4. Inorganic Crystal	d. ( ) materials absorb light and decrease output of light per beta particle.
5. Organic Scintillators	e. ( ) solute molecules interact and dissipate their energy as heat.
6. Liquid Scintillators	f. ( ) causes reduction of probable number of beta particle-solvent molecule interactions.
7. Liquid Scintillation Medium	g. ( ) molecules of fluor and solution interact, absorbing energy before it can be emitted as light.
8. Plastic Scintillator	h. ( ) a reduction of the efficiency of the energy transfer process.
9. Photomultiplier Tube	i. ( ) a secondary scintillant interaction.
10. Photocathode	j. ( ) series of electrodes that emit electrons.
11. Dynode	k. ( ) emits electrons when struck by light.
12. Quenching	l. ( ) a position-emitting, bimetallic secondary solute.
13. Chemical Quenching	m. ( ) combines photoelectron tube with electron-multiplying device.
14. Dilution Quenching	n. ( ) a solid organic scintillant solution.
15. Self Quenching	o. ( ) 5 grams of p-terphenyl dissolved in one liter of dioxane.
16. Color Quenching	p. ( ) allows source to be placed directly in them.
17. Optical Quenching	q. ( ) generally aromatic hydrocarbons containing benzene rings.
18. Light Guide	r. ( ) alkali halides that form regular crystal lattices.
19. Fluor	s. ( ) luminescent material.
	t. ( ) absorption of energy by a substance, followed by re-emission of light (re-emission occurs within 10^{-8} seconds).
	u. ( ) absorption of energy by a substance, followed by re-emission of light (the process takes place after 10^{-3} seconds).

B. Multiple Choice.

Choose the best answer or answers to the following questions; circle the correct answer(s).

20. The four types of scintillators are –
    a. Solid, liquid, gaseous, colloidal.
    b. Organic, inorganic, crystal, liquid.
    c. Liquid, plastic, organic, and inorganic.
    d. Liquid, plastic, gaseous, and organic.
21. A typical liquid scintillation medium is –
    a. One liter of POPOP dissolved in three liters of dioxane.
    b. BBOT dissolved in xylene.
    c. PPO dissolved in toluene.
    d. Five grams of p-terphenyl dissolved in one liter of benzene.
22. Quenching –
    a. Sometimes may increase counting efficiency.
    b. Always decreases counting efficiency.
    c. Occurs rarely.
    d. Increases counting efficiency of certain substances.
23. The coincident light logic is utilized in LSC because it –
    a. Creates thermal noise inside the photomultiplier tube.
    b. Increases the efficiency of a liquid scintillation counter.
    c. Lowers background radiation.
    d. Occurs in one photomultiplier tube at a time.
24. Sample vials should be made of –
    a. A translucent high-Z material.
    b. Ordinary glass.

c. Low-potassium glass.

d. A light-reflecting material.

C. General Descriptive and Graphic Problems.

25. Sketch a photomultiplier tube, and label its components.

26. Describe the flow of electrons through a photomultiplier tube. Include a description of the typical gain of the tube, and describe the relationship of the gain to –

- The number of electrons emitted by the photocathode.
- The quantity of light incident on the photocathode.

(Continue answer for Test item #26 on the next page.)

(Continue answer for Test item #26 here.)

27. Describe the interaction of energetic electrons when a beta-emitting radionuclide is placed in the liquid scintillation medium.

28. Draw a block diagram of a typical liquid scintillation counter, and label its components.

29. List the five characteristics of a primary solvent.

30. Describe the uses of the second solvent and of the primary and secondary solutes.

31. List and describe two methods for the determination of counting efficiency.

NUCLEAR TECHNOLOGY

MODULE 22-6

NEUTRON ACTIVATION ANALYSIS

ADVANCED RADIONUCLIDE ANALYSIS

COURSE 22

CORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 22 ADVANCED RADIONUCLIDE ANALYSIS

MODULE 22-1	Survey of Methods Used in Radionuclide Analysis (Sample Collection & Analysis)
MODULE 22-2	Gamma Spectrometry
MODULE 22-3	Alpha Spectrometry
MODULE 22-4	Beta Spectrometry
MODULE 22-5	Radiochemical Methods of Analysis by Liquid Scintillation Counting
MODULE 22-6	Neutron Activation Analysis
MODULE 22-7	Determination of Specific Nuclides (In the Environment)

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INTRODUCTION

Neutron activation analysis (NAA) is an analytical tool that can be used either qualitatively or quantitatively. In a qualitative mode, it is employed by the researcher to identify the elements that are present in a sample. Used in a quantitative mode, it indicates the amount of each element in a sample.

NAA is a highly sensitive procedure that can detect as little as 10^{-9} gram of some elements for each gram of sample analyzed. This is a sensitivity of one part per billion.

Essentially, NAA is a nondestructive analytical technique, since only very minute quantities of sample material are required. The sample is not damaged and is usually intact after analysis. Crime laboratories employ NAA for the analysis of samples that must be offered as evidence and that cannot be destroyed. It can be used to analyze Ming vases, Roman coins, or medieval weapons without destroying their value as museum specimens. The technique often permits the detection of art forgeries, since the chemical composition of an original work of art can be compared to that of the suspect piece.

NAA permits the simultaneous detection of many different elements. There is no need for tedious testing and retesting. In most cases, the technique is quick, highly sensitive, and accurate.

NAA does have some limitations in chemical analysis. It is not capable of analyzing all elements. Some elements, such as iron, are very difficult to detect; whereas others, such as lead and carbon, cannot be detected at all. Because the sensitivity of an element to NAA is a function of the nuclear composition of an element, not of its atomic

properties, the periodic table offers no clue as to whether or not an element can be detected. For example, manganese is easy to detect, and iron, the element next to it in the table, is difficult to detect.

NAA can detect elements present in a sample, but it cannot analyze how these elements are chemically bound. In an analysis of sediment that contains mercury, NAA would reveal only that some mercury is present in the sample. NAA would not indicate whether the mercury exists as an element, as an inorganic salt, or as an organic compound.

Despite these disadvantages, NAA remains an important analytical technique.

MODULE PREREQUISITES

The student should have completed a course in basic algebra and Course 11, "Radiation Detection and Measurement."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Write typical reaction equations for the (n,γ), (n,α), and $(n,2n)$ reactions of several elements.
2. Describe the methods of quantitative and qualitative neutron activation analysis.

3. Given all necessary variables, calculate the expected activity that results from the neutron irradiation of a standard.
4. Given all necessary variables, calculate the quantity of a trace element in a sample.
5. List three limitations of neutron activation analysis.
6. Given a gamma spectrometer and an irradiated foil, determine the amount of trace impurities in the foil.

THEORY OF NEUTRON ACTIVATION ANALYSIS

In neutron activation analysis, the sample to be analyzed is exposed to a stream of neutrons produced by a nuclear reactor, isotope sources, or particle accelerator; and certain elements in the sample are transformed by this treatment into radioisotopes. The gamma radiation emitted by the neutron-activated sample is analyzed instrumentally to determine the energy of the gamma rays and the intensity and rate of decay of the radiation. Since each radioisotope has a characteristic half-life and set of gamma-ray energies, the identity and amount of the neutron-activated radioisotopes can be determined by comparison to standards that have been activated under identical conditions. The advantages of this technique arise mainly from its extreme sensitivity.

The most common neutron interaction with matter is the neutron-gamma interaction (n,γ) described by Equation 22-6-1.

$$ {}^{A}_{Z}X + {}^{1}_{0}n \rightarrow {}^{A+1}_{Z}X + \gamma \qquad \text{Equation 22-6-1 (Equation = Eq.)} $$

In this reaction, the incident neutron cannot transfer enough energy to the nucleus to cause it to emit a particle, so a gamma ray is emitted.

Other interactions are possible but rarely occur. Intermediate-energy neutrons can transfer enough kinetic energy to the nucleus to cause the emission of either a

proton or an alpha particle. A neutron-proton interaction (n,p) is described by Equation 22-6-2, and a neutron-alpha interaction (n,α) is presented in Equation 22-6-3.

$$ {}^{A}_{Z}X + {}^{1}_{0}n \rightarrow {}^{A}_{Z-1}X + {}^{1}_{1}p \qquad \text{Eq. 22-6-2} $$

$$ {}^{A}_{Z}X + {}^{1}_{0}n \rightarrow {}^{A-3}_{Z-2}X + {}^{4}_{2}He \qquad \text{Eq. 22-6-3} $$

A fast neutron can transfer sufficient incident energy to cause the emission of two particles (Eq. 22-6-4). This is called an (n,2n) reaction. NAA is applied to all of these characteristic neutron interactions, but usually is concerned with the (n,γ) interaction.

$$ {}^{A}_{Z}X + {}^{1}_{0}n \rightarrow {}^{A-1}_{Z}X + {}^{1}_{0}n + {}^{1}_{0}n \qquad \text{Eq. 22-6-4} $$

Figure 22-6-1 is a simple, three-part schematic that explains how NAA works. When the sample is irradiated by a neutron, the nucleus can capture it. This process makes the nucleus radioactive. It can decay, having its own half-life, and is accompanied by the emission of a gamma ray. The gamma-ray energy of a radionuclide is its "signature"; and by an analysis of this energy, the identity of the nuclide can be determined. It should be noted that the gamma ray in Equation 22-6-1 is not the gamma ray detected in NAA. The gamma of Equation 22-6-1 comes off in approximately 10^{-12} seconds after n capture. The gamma rays of NAA (such as those illustrated in Figure 22-6-1) are emitted with the $t_{\frac{1}{2}}$ (half-life) of the radioactive nuclide.

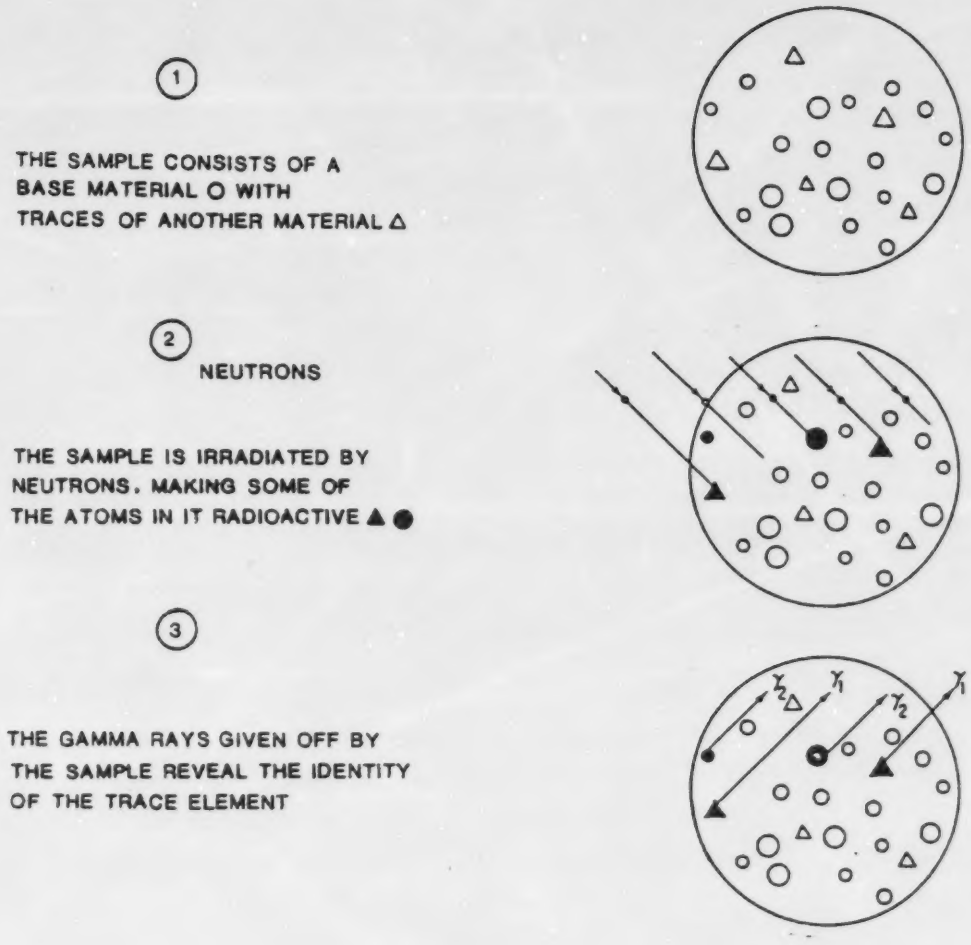

1
THE SAMPLE CONSISTS OF A BASE MATERIAL O WITH TRACES OF ANOTHER MATERIAL Δ
2
NEUTRONS
THE SAMPLE IS IRRADIATED BY NEUTRONS, MAKING SOME OF THE ATOMS IN IT RADIOACTIVE ▲ ●
3
THE GAMMA RAYS GIVEN OFF BY THE SAMPLE REVEAL THE IDENTITY OF THE TRACE ELEMENT
γ2
γ1
γ2
γ1

Figure 22-6-1. Schematic view of activation analysis.

METHODS OF ANALYSIS

Qualitative analysis, which reveals the trace elements in a sample, usually is performed as a preliminary to the quantitative mode of NAA.

The systematic identification of a nuclide is accomplished when the sample is bombarded by neutrons and counted. Gamma-ray energy and half-life, which are characteristic for each nuclide, then are used to identify the nuclide in the sample.

Before a quantitative analysis can be conducted, the chemical identity of the trace elements in the sample must be known. The sample and known amounts of the elements are irradiated. Both the sample and the known quantities (called the "standards") are counted. Equation 22-6-5 allows the determination of the amount of material in the sample (m_x).

$$\frac{C_x}{m_x} = \frac{C_s}{m_s} \qquad \text{Eq. 22-6-5}$$

where:

m_x = amount (in grams) of element in sample.
m_s = amount (in grams) of element in standard.
C_x = counts per minute (cpm) of sample.
C_s = counts per minute (cpm) of standard.

EXAMPLE

Given: A 5-microgram standard of Co in a 1.0-g sample of steel. The ^{60}Co count rate in the standard = 2,000 cpm, and the ^{60}Co count rate in the steel sample = 5,000 cpm.

Find: The concentration of Co in the steel (g Co per g of steel).

Solution: Use Eq. 22-6-5:

$$\frac{C_x}{m_x} = \frac{C_s}{m_x}$$

$C_x = 5{,}000$ cpm

$C_s = 2{,}000$ cpm

$m_s = 5 \times 10^{-6}$ g

Substitution:

$$\frac{5{,}000}{m_x} = \frac{2{,}000}{5 \times 10^{-6}}$$

$$m_x = \frac{(5 \times 10^{3})(5 \times 10^{-6})}{2 \times 10^{3}}$$

$$m_x = 1.25 \times 10^{-5} \text{ g}$$

Concentration of Co in steel = 1.25×10^{-5} g Co per g of steel, or 12.5 ppm Co

If the element has a short half-life, decay during counting time cannot be ignored; and all count rates must be reduced to a common time by use of Equation 22-6-6.

$$C_{t_1} = C_x e^{\lambda t} \qquad \text{Eq. 22-6-6}$$

where:

C_t = counts per minute at time t_1.

C_x = counts per minute at time t.

e = 2.718 (natural log base).

λ = decay constant of the radionuclide = $\ell n 2 / t_{\frac{1}{2}}$.

t = decay time from t_1 to t.

EXAMPLE

Given: A 5-microgram standard of ^{28}Al. A 1.0-g sample of steel. At 12:00, the count rate of the ^{28}Al standard was 2,000 cpm; and at 12:05, the count rate of the ^{28}Al in the steel was 5,000 cpm.

Find: The concentration of ^{28}Al in steel.

Solution: NOTE: Because ^{28}Al has a short half-life ($t_{\frac{1}{2}}$ = 2.24 min), decay between 12:00 and 12:05 cannot be ignored, as it was in the first example. Correct all count rates to a common time (for example to 12:00). Use Equation 22-6-6:

$$C_s = 2{,}000 \text{ cpm}$$

$$C_x = 5{,}000\ e^{\lambda t}$$

$$= 5{,}000\ e^{\frac{(0.69315)\ 5 \text{ min}}{2.24 \text{ min}}} = 5{,}000\ (4.70)$$

$$C_x = 23{,}491$$

From Equation 22-6-5 –

$$\frac{23{,}491}{m_x} = \frac{2{,}000}{5 \times 10^{-6}}$$

$$m_x = 5.87 \times 10^{-5} \text{ g}$$

The concentration is 5.87×10^{-5} g Al per g of steel, or 58.7 ppm Al.

PRELIMINARY CALCULATIONS

Some of the characteristics used to identify an unknown radionuclide are the nuclide's half-life, the type of radiation emitted from the nuclide, and the energy of the emitted

radiation. NAA considers all of these identifying properties.

Frequently NAA permits the identification of the trace elements present in a sample. Before a sample is irradiated, safety precautions demand that the expected activities of the chief elements of the sample must be calculated. These activities also must be known in order that they can be taken into account when the sample is counted and the trace-element activity is filtered out. If, for example, a sample of seawater must be analyzed for traces of gold, the major activity after neutron radiation would be the change to ^{24}Na from ^{23}Na and to ^{38}Cl from ^{37}Cl. The experimenter must wait several days for these major activities to decay before starting to count for gold. If the sample is counted too soon, the activity from ^{198}Au will be hidden by the activities of the major elements.

The expected activity of a sample can be calculated from Equation 22-6-7.

$$A = N\sigma\phi\ (1-e^{-\lambda t}) \qquad \text{Eq. 22-6-7}$$

where:

- A = activity of the sample in dps.
- N = number of atoms in a sample.
- σ = cross section in cm^2.
- ϕ = neutron flux in neutrons/cm^2/sec.
- λ = decay constant of the radionuclide = $\ell n2/t_{\frac{1}{2}}$.
- t = the length of irradiation (the units of λ and t should be the same).

The disintegrations per minute of the radionuclide undergoing decay can be calculated if this equation is solved for activity.

EXAMPLE

Given: A 1-g piece of Al to be irradiated for 1 min in a flux of 2×10^{12} n/m^2/sec. The (n,γ) cross section of ^{27}Al is 0.233×10^{-24} cm^2. ^{28}Al half-life is 2.24 min.

Find: The disintegration rate of the ^{28}Al following irradiation.

Solution: Utilize Equation 22-6-7:

$$N = \frac{1.0\ g}{26.98\ g\ mole^{-1}} \cdot 6 \times 10^{23}\ atoms\ mole^{-1}$$

$$N = 2.22 \times 10^{22}\ atoms\ of\ ^{27}Al$$

$$\sigma = 0.233 \times 10^{-24}\ cm^2$$

$$\phi = 2 \times 10^{12}\ n\ cm^{-2}\ sec^{-1}$$

$$(1-e^{-\lambda t}) = \left(1 - e^{-\frac{0.69315}{2.24} \cdot 1.00}\right) = 0.266$$

$$A = N\sigma\phi\ (1-e^{-\lambda t})$$

$$= (2.22 \times 10^{22})(0.233 \times 10^{-24})(2 \times 10^{12})(0.266)$$

$$A = 2.75 \times 10^{9}\ dps$$

Disintegrations per minute (A) is related to counts per minute by the following equation:

$$A = \frac{C}{\varepsilon\ (B.R.)} \qquad \text{Eq. 22-6-8}$$

where:

A = activity in disintegrations per minute.
C = count rate (cpm).
ε = counting efficiency.
B.R. = branching ratio.

NOTE: In Equation 22-6-8, the units of A can be dps, in which case the count rate must be in consistent units (cps).

EXAMPLE	
Given:	Disintegration rate (2.75×10^9 dps) found in Example 3.
Find:	Count rate for the 1.78-MeV gamma ray that has a counting efficiency of 0.3% and a branching ratio of 100%.
Solution:	Equation 22-6-8:

$$A = 2.75 \times 10^9 \text{ dps}$$

$$\varepsilon = 0.3\% = 0.003$$

$$\text{B.R.} = 100\% = 1.0$$

$$2.75 \times 10^9 = \frac{C}{(0.3 \times 10^{-2})(1.00)}$$

$$C = 8.25 \times 10^6 \text{ cps}$$

EXAMPLE	
Given:	The previous examples, 30 minutes after irradiation.
Find:	Count rate of the Al sample.
Solution:	Method 1: Solution from count rate data alone:

$$C_o = C\, e^{\lambda t}$$

where:

C = count rate at time t (30 min)

C_o = count rate at time 0

t = the time between C and C_o

λ = decay constant of the ^{28}Al

$$C = 8.25 \times 10^6\, e^{\frac{0.693}{2.24} \cdot 30}$$

$$= 8.25 \times 10^6\, (9.3 \times 10^{-5})$$

$$C = 767 \text{ cps}$$

Method 2: Solution from disintegration rate data:

$$A_o = A\, e^{-\lambda t}$$

$$= 2.75 \times 10^9\, e^{-\frac{0.693}{2.24} \cdot 30}$$

$$= (2.75 \times 10^9)(9.3 \times 10^{-5})$$

$$A_o = 2.56 \times 10^5 \text{ dps}$$

Equation 22-6-8 and the E and B.R. given in Example 3 yield the following:

$$2.56 \times 10^5 = \frac{C}{(0.3 \times 10^{-2})(1)}$$

$$C = 767 \text{ cps}$$

THE PRACTICE OF NEUTRON ACTIVATION ANALYSIS

In the most common type of activation analysis, the neutron bombardment of a sample is performed in a nuclear reactor, where the neutrons that strike the target atoms have been slowed down so that they have very little energy

of motion. In this case, the usual reaction between the target atoms and a neutron results in the capture of the neutron; this reaction creates a nucleus that has an atomic weight of one more unit than it started with. Thus, for sodium as found in nature (symbol ^{23}Na), the following reaction occurs:

sodium-23 + a neutron → radioactive sodium-24 + gamma rays

The "Swimming Pool" reactors frequently are utilized for neutron activation analysis. In this kind of reactor, the rods that contain nuclear fuel are held by a framework at the bottom of a deep pool of water. The water serves the purpose of a shield for the protection of workers and slows down neutrons in order that they are more likely to interact with the target atoms.

The sample and a standard (or "comparator") are placed into the reactor. The standard is similar in form and composition to the sample to be measured, but contains a known quantity of the element to be determined.

The steps used for the analysis are as follows: (1) The sample and the standard are placed together into the reactor and bombarded with neutrons. (2) The sample and the standard are removed, and the radioactivity produced by the sample is measured. (3) A comparison is made between the radioactivity of the sample and the standard, and the amount of the element in the sample is calculated as a proportion from Equation 22-6-9.

$$\frac{\text{Radioactivity in sample}}{\text{Radioactivity in standard}} = \frac{\text{Quantity of element in sample}}{\text{Quantity of element in standard}} \qquad \text{Eq. 22-6-9}$$

SAMPLE AND STANDARD PREPARATION

A sample and a standard are prepared as follows: (1) A small portion of sample material is scraped from a source. (2) The sample is weighed on an accurate balance (Figure 22-6-2). (3) The sample is placed into a piece of quartz tubing about an inch in length and a quarter inch in diameter, and the tube ends are sealed with an oxygen-gas torch. (4) A portion of standard material is prepared and sealed in the same manner but in a slightly longer tube (in order that the sample can be distinguished from the standard). (5) Both tubes are enclosed in a sealed can (Figure 22-6-3), which is placed in the reactor for irradiation. (Plastic tubes can be used — their open ends melted shut with a soldering iron.)

STEPS IN NAA

The simplest application of NAA is the quantitative analysis of a sample in order that a trace element in it can be identified. As an illustration of this method, sodium (Na) as a trace element in a sample of plastic can be measured by performance of the following eight-step procedure. (In this method, after the number of radioactive atoms created, N_{24}, is measured, the number of target atoms can be calculated.)

Step 1. Weigh a sample and a standard in quartz tubes (Figure 22-6-2).

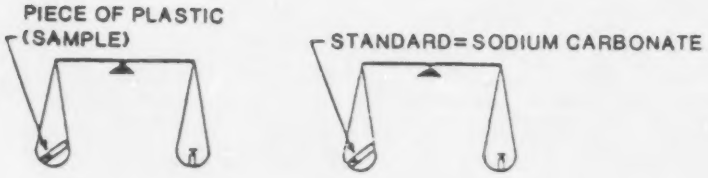
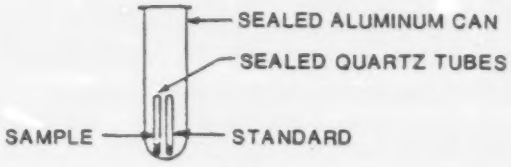


Figure 22-6-2. Weighing of sample and standard.

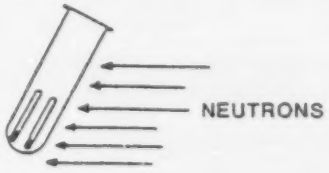
Step 2. Seal tubes in package for reactor irradiation (Figure 22-6-3).

Figure 22-6-3.
Tubes sealed for
reactor irradiation.



Step 3. Bombard with neutrons for about 3 hours in a reactor (Figure 22-6-4).

Figure 22-6-4.
Neutron bombardment.



Step 4. Remove sample and standard from tubes, and place in separate plastic containers to measure gamma rays (Figure 22-6-5).

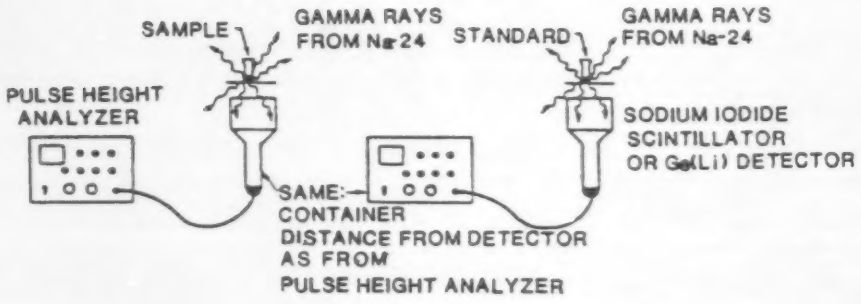

SAMPLE
GAMMA RAYS FROM Na-24
STANDARD
GAMMA RAYS FROM Na-24
PULSE HEIGHT ANALYZER
SODIUM IODIDE SCINTILLATOR OR Ge(Li) DETECTOR
SAME: CONTAINER DISTANCE FROM DETECTOR AS FROM PULSE HEIGHT ANALYZER

Figure 22-6-5. Measurement of gamma rays.

Step 5. Obtain gamma-ray spectrum for sodium-24 in both sample and standard (Figure 22-6-6).

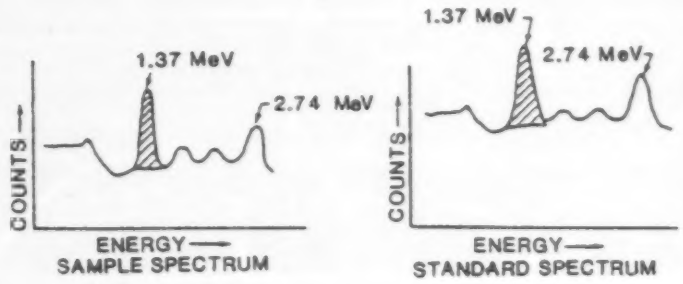


Figure 22-6-6. Gamma-ray spectra.

Step 6. Use standard to calculate 1.37-MeV gamma rays counted per minute per gram of sodium (c/m/gNa).

$$c/m/gNa = \frac{\text{counts/minute measured in 1.37 peak (shaded area in Figure 22-6-6)}}{\text{grams of sodium known to be in standard (Step 1)}}$$

Step 7. Use c/m/gNa and 1.37-MeV gamma rays counted per minute in sample to calculate grams of sodium in sample.

$$\text{grams Na in sample} = \frac{\text{counts/minute measured in sample}}{\text{c/m/gNa (Step 6)}}$$

Step 8. Calculate percent sodium in sample.

$$\% \text{ sodium} = \frac{\text{grams sodium in sample (Step 7)}}{\text{weight of sample (Step 1)}} \times 100$$

After the preliminary calculations of major activities have been performed, the experimenter must ensure that σ, B.R., λ, E, t, and ϕ are the same in both the sample and in the standard. The value of m_x is estimated, and m_s is made to approximate the mass of m_x. The sample and the standard are irradiated for the same length of time in an identical neutron flux. The sample and standard then are counted. Because the absolute decay rate A of the nuclide is known, the rate can be extended to the time when irradiation was discontinued. The quantity of the target nuclide can be estimated with Equations 22-6-7 and 22-6-8. The amount of target nuclide can be determined exactly with the ratio in Equation 22-6-5.

EXAMPLE

Refer to step-by-step calculations:

Given: Mass of standard = 1.0 mg Na_2Co_3.
Mass of sample = 2.5 g.
Half-life of ^{24}Na = 15 hours.
End of bombardment = 00:00 (midnight).
Standard count at 12:00 = 5,000 counts per 60-minute count in the 1.37-MeV peak.

Sample count at 15:00 = 3,000 counts per 120 min count in the 1.37-MeV peak.

Find: % Na in the sample.

Solution: (1) Count rates: extrapolate all counts back to midnight.

$$C_s = \frac{5{,}000 \text{ counts}}{60 \text{ minutes}} e^{\lambda t}$$

$$= 83.3 \text{ cpm } e^{\frac{0.693}{15} \cdot 12.5}$$

(The value t = 12.5 is a result of the assumption that the count rate 83.3 cpm occurs halfway through the count; therefore, t = 12 hr + 0.5 hr).

$$C_s = 83.3 \ (1.78) = 148 \text{ cpm}$$

$$C_x = \frac{3{,}000}{120} e^{\frac{(0.693)(16)}{15}}$$

$$= 25 \text{ cpm } (2.09)$$

$$C_x = 52.4 \text{ cpm}$$

(2) Na is the standard: Multiply 1 mg by the fraction of Na in Na_2Co_3.

$$m_s = (1 \times 10^{-3} \text{ g } Na_2Co_3)\frac{2(22.99)}{60}$$

$$= (1 \times 10^{-3})(0.766)$$

$$m_s = 7.66 \times 10^{-4} \text{ g}$$

(3) Determine the amount in grams of Na in the sample:

$$\frac{C_x}{m_x} = \frac{C_s}{m_s}$$

$$\frac{52.4 \text{ cpm}}{m_x} = \frac{148 \text{ cpm}}{7.66 \times 10^{-4} \text{ g}}$$

$$m_x = 2.71 \times 10^{-4} \text{ g}$$

(4) The % Na is $\frac{2.71 \times 10^{-4} \text{ g}}{2.5 \text{ g}} \times 100 = 0.011\%$

LIMITATIONS OF NEUTRON ACTIVATION ANALYSIS

One problem with NAA is that not all elements are readily activated, and some cannot be activated at all. Table 22-6-1 provides a partial list of some of the more readily activated nuclides.

TABLE 22-6-1.

PARTIAL LIST OF MORE READILY-ACTIVATED NUCLIDES.

Target Nuclides	% Natural Abundance	Isotope Produced	Half-Life	Isotopic Cross Section (barns)	For $\phi = 10^5$ Approximate Sensitivity (mg nuclide)
^{45}Sc	100	^{46}Sc	84d	15	2.0
^{51}V	99.76	^{52}V	3.77m	4.5	
^{55}Mn	100	^{56}Mn	2.58h	13.3	0.6
^{59}Co	100	^{60m}Co	10.5m	18	
^{63}Cu	69.09	^{64}Cu	12.9h	4.5	7.0
^{65}Cu	30.09	^{66}Cu	5.1m	2.3	
^{75}As	100	^{76}As	26.3h	4.3	2.0
^{79}Br	50.54	^{80}Br	18m	8.5	
		^{80m}Br	4.5h	2.9	5.0
^{81}Br	49.46	^{82}Br	35.7h	3	
^{108}Pd	26.71	^{109}Pd	13.6h	12	5.0
^{107}Ag	51.82	^{108}Ag	2.4m	40	
^{109}Ag	48.18	^{110}Ag	249d	2.8	
		^{110}Ag	24s	82	
^{115}In	95.72	^{116m}In	54m	150	0.1
		^{116}In	14s	50	

^{121}Sb	57.25	^{122}Sb	2.8d	6	4.0
^{123}Sb	42.75	^{122m_2}Sb ^{124m_1}Sb	21m 1.3m	30 30	
^{127}I	100	^{128}I	25m	6.4	2.0
^{139}La	99.911	^{142}La	40.2h	8.9	2.0
^{141}Pr	100	^{142}Pr	19.2h	11	2.0
^{152}Sm	26.72	^{153}Sm	46.7h	220	0.6
^{151}Eu	47.82	^{152}Eu	9.3h	1700	0.03
^{159}Tb	100	^{160}Tb	73d	46	4.0
^{164}Dy	28.18	^{165}Dy ^{165m}Dy	2.3h 75s	800 2000	0.03
^{165}Ho	100	^{166}Ho	27.2h	64	0.4
^{169}Tm	100	^{170}Tm	127d	125	2.0
^{174}Yb	31.84	^{175}Yb	4.2d	60	2.0
^{175}Lu	97.41	^{176m}Lu	3.7h	18	1.0
^{186}W	28.41	^{187}W	24.0h	40	3.0
^{185}Re	37.07	^{186}Re	90h	110	0.7
^{187}Re	62.93	^{188}Re	17h	70	0.6
^{191}Ir	37.3	^{192}Ir ^{192m}Ir	74d 1.4	730 250	0.4
^{193}Ir	62.7	^{194}Ir	19h	130	0.3
^{197}Au	100	^{198}Au	64.8h	96	3.0

Another problem arises when identical gamma energies are emitted by different elements. The following are some of the element pairs that emit identical gamma energies: ^{203}Hg and ^{22}Se, ^{56}Mn and ^{27}Mg, and ^{65}Zn and ^{59}Fe. When this situation occurs, the elements can be differentiated by calculating the half-life of the element.

Sometimes the gamma energies of a trace element are buried by the Compton-scattered gammas of other activities in the matrix. The problem of hidden gamma energies frequently occurs in biological samples, since ^{24}Na, a commonly-occurring biological element, emits many Compton-scattered gamma rays.

EXERCISES

1. List all the possible radioactive products resulting from the bombardment of ^{27}Mg by neutrons. Consider the following possible reactions: (n,p), (n,γ), (n,2n), and (n,α).
2. NAA will be used to detect tellurium (Te) in an environmental sample.
   - Make a list, considered as a function of half-life, of all the possible gamma rays produced by the irradiation of a piece of natural tellurium by neutrons. Assume that the only reaction occurring is the (n,γ) reaction.
   - Make a list, considered as a function of gamma-ray energy, of all the gamma rays that can be detected two weeks after irradiation. Include the branching ratio of each gamma ray.
   - Determine which of the gamma rays of tellurium should be selected for NAA.
3. Calculate the activity (dps) of 1.0 gram of aluminum 10 minutes after it has been irradiated in a flux of 2×10^{12} neutrons/cm^2/sec for fifteen seconds. The decay constant (λ) and cross section (σ) should be obtained from references.
4. Calculate the number of micrograms of manganese in one gram of aluminum. Use the following data:
   - A 1.0-gram sample of Al was irradiated by neutrons. Irradiation stopped at 10:00 o'clock.
   - During the irradiation of Al, a 1.0-gram Mn standard was also irradiated.
   - The count rate of the Mn in the sample at 10:21 was 12,381 cpm. The count rate of the Mn standard at 10:15 was 15,382 cpm.

MATERIALS

The use of a research reactor and access to a gamma-sensitive counter. (A list of research reactors is given in the nuclear Program Planning Guide.)

~1 gram of Al foil (commercial quality).

100-microgram Mn standard. (For preparation, refer to Step 3 in Procedures.)

PROCEDURES

DETERMINATION OF MANGANESE IN COMMERCIAL ALUMINUM FOIL

1. Secure time on a research reactor and access to gamma-sensitive counting equipment.
2. Weigh out 1 gram of Al foil. This mass equals m_x.
3. Make a Mn standard of 100 micrograms Mn. To accomplish this, weigh out 14 milligrams of MnO_2 and dissolve it in dilute HNO_3. Dilute the solution with H_2O until its volume is 100 milliliters, and pipette out 1 milliliter.
4. Irradiate the standard and the sample for one minute in a neutron flux of 2×10^{12} $n/cm^2/sec$. WARNING: Al foil presents a radiation hazard due to the presence of ^{28}Al for 30 minutes after the end of neutron bombardment.
5. Let ^{28}Al decay for at least 30 minutes.
6. Count the sample and the standard once every 5 minutes for 30 to 90 minutes after irradiation; then count once every 15 minutes for the next 2 hours.
7. Plot the log cpm of the 0.84-MeV gamma ray against the time of the count.

8. Observe the decay in the sample of ^{27}Mg produced by the ^{27}Al (n,p) ^{27}Mg reaction.
9. Observe the ^{56}Mn decay. Extrapolate the activity in both the standard and the sample to their end-of-bombardment time. These activities will give C_s and C_x.
10. Calculate the mass in micrograms of Mn in the Al. Use Equation 22-6-5.

DATA TABLE

DATA TABLE 22-6-1

1. Weight of Al foil: m_1 = _____ g
2. Mn standard:
   Concentration of Mn stock solution:
   _____ g/ _____ ml = _____ g/ml = A
   Volume irradiated:
   _____ ml = B
   Mass of standard = A·B = _____ g = m_s
3. Times of end of bombardment (EOB):
   Al foil _____ Mn standard _____
4. Count data:

Sample	Time of Count	ΔT of Count	Net cpm

(Data Table 22-6-1 continued)

Sample	Time of Count	ΔT of Count	Net cpm

5. Count rate of ^{56}Mn at end of bombardment (EOB):

   Sample = _____ cpm = C_x

   Standard = _____ cpm = C_s

6. Grams of Mn in sample:

$$m_x = m_s \frac{C_x}{C_s} = \_\_\_\_\_ \text{ g}$$

7. Concentration of Mn in Al foil = m_x/m_1

   = _____ gMn/gAl

REFERENCE MATERIALS

Chase, Grafton D. and Rabinowitz, Joseph L. Principles of Radioisotope Methodology. 3rd ed. Minneapolis: Burgess Publishing Company, 1967.

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Guinn, V. P.; Weissberger; and Rossiter, ed. Physical Methods of Chemistry, Volume I, Part III D. New York: Wiley-Interscience, 1972.

Keisch, Bernard. The Atomic Fingerprint: Neutron Activation Analysis. Oak Ridge, TN: United States Energy Research and Development Administration, Technical Information Center, 1972.

Kruger, P. Principles of Activation Analysis. New York: John Wiley & Sons, Inc., 1971.

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TEST

1. Write reaction equations for the (n,γ), (n,α), and $(n,2n)$ reactions on ^{31}P, ^{30}Si, and ^{16}O.

2. Describe the methods of quantitative and qualitative neutron activation analysis.

3. What is the expected activity of ^{76}As produced from 1 microgram of arsenic at the time it comes out of a reactor?

$N = 8 \times 10^{15}$

$\phi = 10^{13}$ neutrons/cm^2/sec

$\sigma = 4.2 \times 10^{-24}$ cm^2

$\lambda = 0.026$

$t = 12$ hours

4. On January 1, 1.58 g of a biological sample containing Co was irradiated along with 15.6 mg of a cobalt standard. The end of bombardment (EOB) was at 12 noon. On February 12, each sample was counted for 1 hour. The counting of the biological sample started at 9 A.M., and the standard was counted at noon. The net counts in the 1.33-MeV gamma peak were as follows:

Sample: 13,581

Standard: 10,318

Calculate the number of grams of Co per gram of biological specimen.

5. The amount of Cu in a piece of steel is to be determined by NAA. The following data has been collected:

	Standard	Steel Sample
Mass	6:1 mg	5.35 g
End of Bombardment	12:30	12:45
Time of Count	12:42	12:52
Δt of Count	3 min	3 min
Counts in the 1.04 MeV γ or ^{66}Cu	5,314	7,815

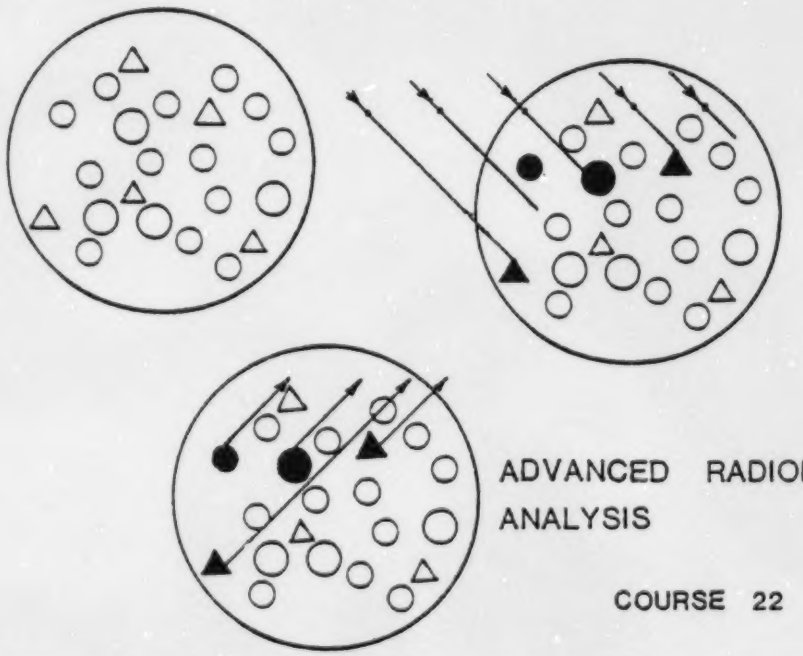
Calculate the number of grams of Cu per gram of steel.

6. List three limitations of neutron activation analysis.

NUCLEAR TECHNOLOGY

MODULE 22-7

DETERMINATION OF SPECIFIC NUCLIDES IN THE ENVIRONMENT.



ADVANCED RADIONUCLIDE ANALYSIS

COURSE 22

ORD CENTER FOR OCCUPATIONAL RESEARCH AND DEVELOPMENT

COURSE MODULES

COURSE 22 ADVANCED RADIONUCLIDE ANALYSIS

MODULE 22-1	Survey of Methods Used in Radionuclide Analysis (Sample Collection & Analysis)
MODULE 22-2	Gamma Spectrometry
MODULE 22-3	Alpha Spectrometry
MODULE 22-4	Beta Spectrometry
MODULE 22-5	Radiochemical Methods of Analysis by Liquid Scintillation Counting
MODULE 22-6	Neutron Activation Analysis
MODULE 22-7	Determination of Specific Nuclides (In the Environment)

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INTRODUCTION

Nuclear analysis can be either quantitative or qualitative. Qualitative analysis is used to identify the nuclides present in a sample; quantitative analysis, to measure the quantity of a nuclide.

Gross alpha and beta counting is the most common type of analysis performed on environmental samples. Frequently, no further analysis is required. The analyses taught in this module generally would be required only on samples that demonstrate a gross alpha or beta activity above permissible limits.

Other than gross alpha and beta analysis, gamma isotopic is the most frequently-used analysis for environmental samples. Gamma analysis provides qualitative and quantitative information simultaneously.

The qualitative analysis of nuclides is based upon their observable chemical and physical properties, including their half-life, the type of radiation emitted, and the energy of the radiation. Actual wet chemical analysis sometimes is performed. These techniques, with the exception of chemical analysis, have been discussed in previous modules. Chemical analysis may be performed when a tentative identification already has been made.

Quantitative analysis involves different strategies entirely and is performed for any of several purposes, most commonly to –

- Measure the decay rate of a source.
- Measure the radiation intensity of a given place.
- Measure the concentration of a specific nuclide.

This module details methods of quantitative analysis and is especially concerned with techniques that a technician can employ to measure the concentration of specific nuclides in the environment. It also brings together the techniques

of qualitative analysis outlined in the previous modules and provides guidelines for the systematic identification and measurement of radionuclides.

MODULE PREREQUISITES

The student should have completed a course in basic algebra; a course in basic chemistry; Course 2, "Radiation Protection I"; and Course 11, "Radiation Detection Systems."

OBJECTIVES

Upon completion of this module, the student should be able to:

1. Differentiate between qualitative and quantitative analysis of radionuclides in environmental samples.
2. List the four general means by which radionuclides can be identified.
3. Use appropriate half-life tables to identify a radionuclide.
4. Specify methods for determining the type of radiation emitted by a radionuclide.
5. List nuclides that could be present in the environment due to the activities of a nuclear power plant or a reprocessing plant.
6. List the complete procedure for determining the con-

centrations of the following:

- ^{60}Co in sediments or soil samples.
- ^{90}Sr in vegetation.
- Tritium in a water sample.

QUALITATIVE IDENTIFICATION OF NUCLIDES

Nuclides can be identified as follows:

- Measurement of their half-life.
- Identification of the types of radiation emitted.
- Measurement of the energy of the radiation.
- Chemical analysis.

Data obtained from any or all of these procedures is compared to the data in the literature to provide identification of the nuclide.

IDENTIFICATION BY HALF-LIFE, EMITTED RADIATION, OR ENERGY

Procedures for determining half-life have not been discussed. Since the method of choice for measuring the half-life of a radioactive nuclide depends upon the magnitude of the half-life to be measured, use of these techniques presupposes that the technician has some idea of the type of nuclide present. This isn't always possible. Measurements of relatively long half-lives also present problems and often are not used when spectroscopic methods are available or when immediate results are required. When a half-life method is employed, the nuclide is identified from tables such as Table 22-7-1 and Table 22-7-2.

TABLE 22-7-1
BETA PARTICLE ENERGIES AND HALF-LIVES

	Half-life						
	< 1 hour	1-6 hours	6-24 hours	1-5 days	5-30 days	30 days-1 year	> 1 year
0-0.3 MeV				^{199}Au	^{191}Os	*^{35}S, *^{45}Ca (2)^{55}Fe, ^{95}Nb ^{103}Ru, ^{203}Hg	*^{3}H, *^{14}C *^{63}Ni, *^{99}Tc (2)^{134}Cs, ^{147}Pm, ^{155}Eu
				^{82}Br, *^{121}Sn (1)^{175}Yb	^{133}Ze, *^{169}Er ^{177}Lu	^{46}Sc, (1)^{59}Fe ^{95}Zr, ^{141}Ce ^{144}Ce, ^{181}Hf *^{185}W	^{60}Co
0.5-0.7 MeV		(3)^{56}Mn, (2)^{65}Ni	(1)^{64}Cu, ^{127}Te (2)^{159}Gd, (1)^{187}W, ^{197}Pt	^{77}As (1)^{105}Rh	^{131}I	(1)^{124}Sb, ^{192}Ir	^{22}Na, ^{85}Kr, ^{90}Sr (1)^{134}Cs, ^{137}Cs
0.7-1.0 MeV	^{69}Zn	^{85m}Kr	(1)^{72}Ga, (1)^{159}Gd	^{198}Au	*^{143}Pr, (1)^{147}Nd	^{170}Tm	*^{36}Cl, *^{204}Tl
1.0-1.5 MeV	(2) ^{38}Cl	*^{31}Si, ^{41}A (2)^{56}Mn, (3)^{65}Ni, ^{97}Nb ^{105}Ru, ^{149}Nd	^{24}Na, ^{109}Pd ^{171}Er	^{99}Mo, (1)^{122}Sb, ^{145}Ce, ^{149}Pm (1)^{186}Re, ^{191}Os, *^{210}B	^{111}Ag	*^{89}Sr	
1.5-3.0 MeV	^{28}Al, ^{76}Ga (2)^{86}Rb, ^{27}Mg	(1)^{56}Mn, (1)^{68}Ni ^{80}Br, ^{139}Ba	(2)^{42}K, (2)^{72}Ga, ^{97}Zr ^{143}Pr, ^{188}Re, ^{194}Ir	^{76}As, *^{88}Y (2)^{122}Sb, ^{166}Ho	*^{32}P, (1)^{86}Rb	^{91}Y, ^{114}In ^{111m}Cd, (2)^{124}Sb	
>3.0 MeV	(1)^{38}Cl, (1)^{88}Rb		(1)^{42}K, (3)^{72}Ga			^{144}Ce/^{144}Pr	^{106}Ru/^{106}Rh

Numbers in parentheses indicate that more than one β-group exists. The most abundant group is numbered (1), the second most abundant (2) and so on.

*Pure β-emitter.

TABLE 22-7-2

GAMMA ENERGIES AND HALF-LIVES

	Half-life						
	< 1 hour	1-6 hours	6-24 hours	1-5 days	5-30 days	30 days-1 year	> 1 year
0-0.3 MeV		(1) ^{133}Ba	^{171}Er, ^{188}Re	^{133m}Xe ^{153}Sm, ^{199}Au	^{133}Xe (2) ^{147}Nd	^{141}Ce, ^{170}Tm (2) ^{181}Hf, ^{203}Hg	^{155}Eu
0.3-0.5 MeV	(2) ^{116m}In	^{87m}Sr	^{69m}Zn, ^{159}Gd	^{105}Rh (2) ^{140}La ^{198}Au	^{51}Cr, ^{131}I	^{7}Be, ^{113}Sn ^{175}Hf (1) ^{181}Hf	^{133}Ba
0.5-0.7 MeV		^{80}Br, ^{97}Nb	^{64}Cu, (β^+)	(1) ^{76}As, (1) ^{82}Br ^{121}Sb	^{52}Mn(β^+) (1) ^{147}Nd	^{58}Co(β^+) ^{103}Ru (1) ^{124}Sb	^{22}Na(β^+) (1) ^{134}Cs ^{137}Cs
0.7-1.0 MeV	(2) ^{88}Rb, ^{27}Mg	(1) ^{56}Mn, ^{105}Ru, (1) ^{132}I	(1) ^{72}Ga, ^{97}Zr	(2) ^{82}Br	(1) ^{52}Mn	(1) ^{46}Sc ^{54}Mn, ^{58}Co, ^{95}Zr, ^{95}Nb	(2) ^{135}Cs
1.0-1.5 MeV	(1) ^{116m}In	^{42}A, ^{65}Ni (2) ^{133}Ba	(1) ^{24}Na	(2) ^{76}As, (3) ^{82}Br	(2) ^{52}Mn, ^{86}Rb	(2) ^{46}Sc, ^{59}Fe ^{65}Zn	^{22}Na, ^{60}Co
1.5-3.0 MeV	^{28}Al, ^{38}Cl (1) ^{88}Rb (3) ^{116m}In	(2) ^{56}Mn	(2) ^{24}Na, ^{42}K (2) ^{72}Ga, ^{142}Pr	(1) ^{140}La		(2) ^{124}Sb	

Only isotopes having relatively simple γ-decay schemes are listed.

Numbers in parentheses indicate more than one γ-group exists. The most abundant group is numbered (1), the second most abundant (2) and so on.

Identification of the type of radiation emitted is often useful, especially as a preliminary step to spectroscopic analysis.

Gamma radiation can be detected by placing a quarter-inch sheet of plastic between the source and a detector. Alpha and beta radiation will be absorbed by the plastic, and only gamma radiation will pass through for detection.

Beta radiation is detected by holding the source two inches or slightly more from the window of a GM tube, placing the quarter-inch plastic between the source and the tube, and observing the activity with and without the plastic. Since alpha radiation will be absorbed by the air, and since the GM tube has an efficiency for gammas of only 1-2%, any increase in radiation will mean that beta radiation is present. Very soft beta radiation may not be detected by this method.

Alpha-particle radiation usually is identified by probing with a scintillation probe equipped with a zinc sulfide screen or by biasing a proportional counter to discriminate against beta and gamma radiation.

Spectroscopic techniques usually are employed in the measurement of radiation energies. Although spectrometry is the fastest and most accurate method, other techniques may be employed.

For alpha particles, the techniques involve the conversion of their range in air to energy. These techniques are described specifically in Module 22-3, "Alpha Spectrometry."

For beta particles, Feather Analysis often is more practical than beta spectroscopy. This technique has been discussed in the text and Procedures section of Module 22-4, "Beta Spectrometry."

Gamma energy can be estimated from the absorption coefficient. Calibrated lead absorbers are placed between the source and the counter, and the activity of the sample is measured for each absorber until the activity has decreased to 50% of the original value. In this way, the half-value layer for lead is known and can be related to energy by means of a graph such as the one depicted in Figure 22-7-1.

Spectrometry is, of course, a better method for measurement of gamma energies, as discussed in previous modules.

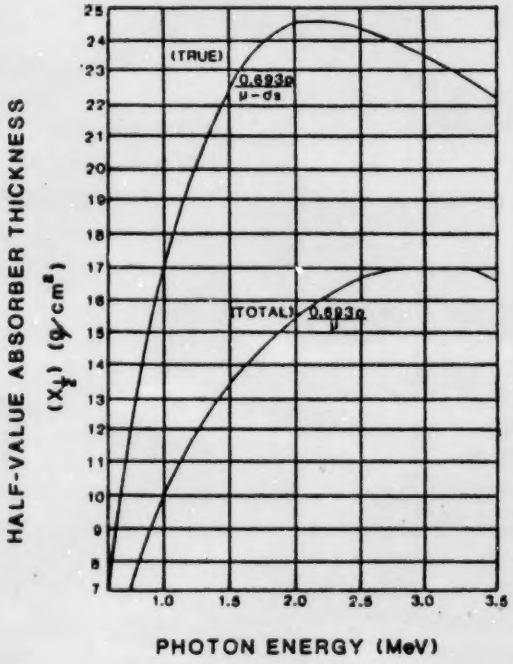

Figure 22-7-1. Half-value layers of lead as a function of proton energy.

RADIOCHEMICAL SEPARATION METHODS

Prior to the counting of a radionuclide, several preparatory steps must be taken to insure proper data collection. These steps include the following:

- Separation of the isotope from any contaminating materials and/or other isotopes.
- Concentration of the activity of the sample to a small volume.
- Rendering of the sample into a form suitable to counting techniques and requirements.

Methods outlined here may accomplish one or more of these steps. In other instances, additional sample preparation may be necessary. The purpose of this section is to provide a general overview of the methods available. Additional detail on any of the methods listed may be found in the reference materials.

When a tentative identification of the nuclide has been made and the source contains a mixture of nuclides, the nuclides can be separated chemically. Various radiochemical techniques are available and include (1) precipitation, (2) solvent extraction, (3) chromatography, and (4) electrochemical separation.

Precipitation

Precipitation as a separatory method is accomplished by causing one component of a solution to drop out of solution as a solid (the precipitate) while leaving all other components of the mixture in the liquid portion, or supernatant. The precipitate then must be separated from the

supernatant solution.

After the precipitation process is complete, the precipitate may be separated from the supernatant by filtration. In this process, both the precipitate and supernatant are added to a filter apparatus similar to that pictured in Figure 22-7-2. The apparatus consists of a filter funnel inserted into a side-arm flask, which will provide the vacuum to carry out the filtration and drying process. A test tube can be installed under the filter funnel to retrieve the supernatant, if desired.

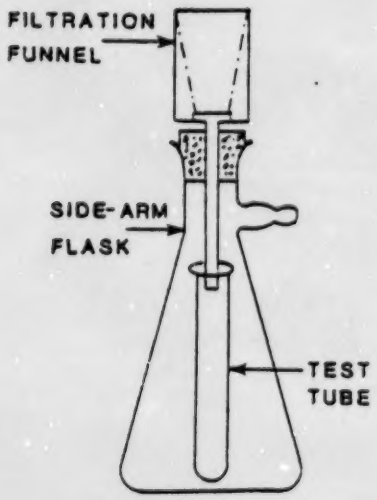

Figure 22-7-2. Assembled filtration apparatus.

The precipitate thus collected on the filter pad of the funnel then can be removed and the sample prepared for counting.

An alternate method of separating precipitate and supernatant is centrifugation. After the precipitation process is completed, the precipitate and supernatant are mixed into suspension and then centrifuged. After centrifugation, the supernatant is transferred to a clean container; and

the precipitate remaining is washed with a small quantity of a suitable liquid, remixed, and centrifuged again. The resulting supernatant is added to the previously-collected supernatant, and the precipitate suspended in a suitable liquid. The suspended precipitate then is transferred to an appropriate sample holder and evaporated to dryness under a heat lamp or similar heat source.

Solvent Extraction

A second method of component separation is solvent extraction. Solvent extraction is based upon the ratio of the equilibrium concentrations of a given solute between two immiscible solvents. A suitable solvent is added to the solution containing the nuclide to be separated in a separatory funnel (Figure 22-7-3), and the contents are then agitated to mix the two solutions and to extract the desired nuclide into the solvent portion of the mixture. The two solutions then are allowed to separate, and the solvent fraction containing the nuclide is removed from the funnel. A portion of the solvent solution may be placed into a planchet and evaporated to dryness in preparation for counting procedures.

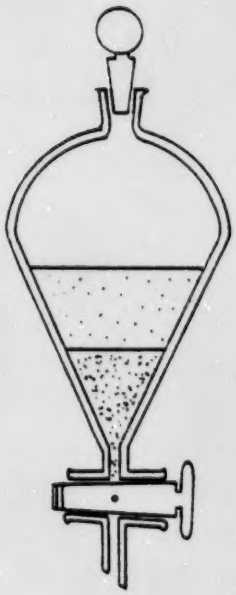

Figure 22-7-3. Solvent extraction apparatus.

Chromatography

Chromatography is a separatory process with many variations. A common feature of all chromatographic methods is that a fluid mobile phase containing the sample to be separated passes over a stationary phase with which the sample may react in a reversible manner. Because of the different relative affinities of each substance for the two selected phases, different components of the sample will be distributed differently between the mobile and stationary phases and, consequently, will move with different speeds. This difference in component mobility enables one to separate each component within the chromatographic apparatus and to collect them separately.

In column adsorption chromatography, the stationary phase is a solid material packed into a vertical glass cylinder or column (Figure 22-7-4). A suitable liquid mobile phase containing the sample is allowed to flow slowly downward through the column. The relative affinities of the sample components for each of the two phases will separate the components within the column. The collected eluate fraction containing the desired nuclide then can be prepared for counting. Column partition chromatography is basically the same process as column adsorption chromatography, except that the solid stationary phase first is wetted with a suitable liquid before introduction of the mobile phase. Separation of the sample components is then a function of the component's relative solubilities in each of the two liquids.

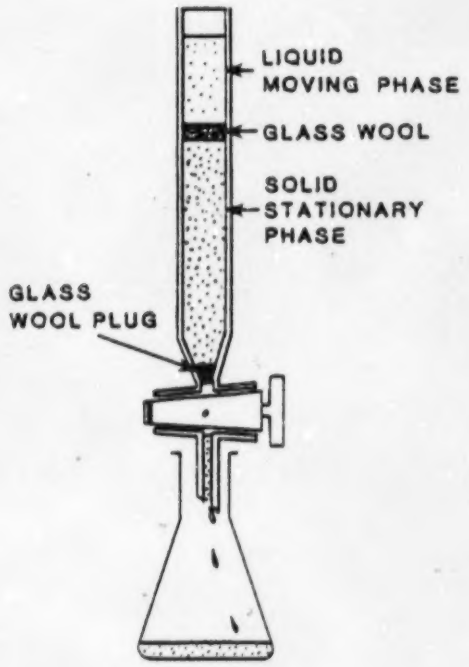

Figure 22-7-4. Column chromatography apparatus.

Paper chromatography is similar to column chromatography in that the paper method also is concerned with the movement of a liquid mobile phase over a solid stationary phase. Paper chromatography can be performed by either ascending or descending methods – the only difference being the direction of travel of the mobile phase (Figures 22-7-5 and 22-7-6). In paper chromatography, the solid stationary phase is represented by a strip of filter paper. This strip of paper will have a starting line drawn across it near one end of the strip, and a solution containing the substances to be separated is "spotted" on that line. A solvent suitable to the components contained in the sample is selected and allowed to travel along the filter-paper strip. In ascending chromatography (Figure 22-7-5), this is accomplished by suspending the strip in such a way as to submerge the end near the starting line in a reservoir of the solvent and allowing the solvent to travel upward along the strip. In descending paper chromatography (Figure 22-7-6), the end near the starting line is placed in a solvent reservoir; and the solvent allowed to travel downward along the strip.

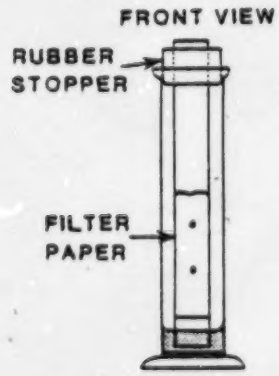

Figure 22-7-5. Ascending paper chromatography.

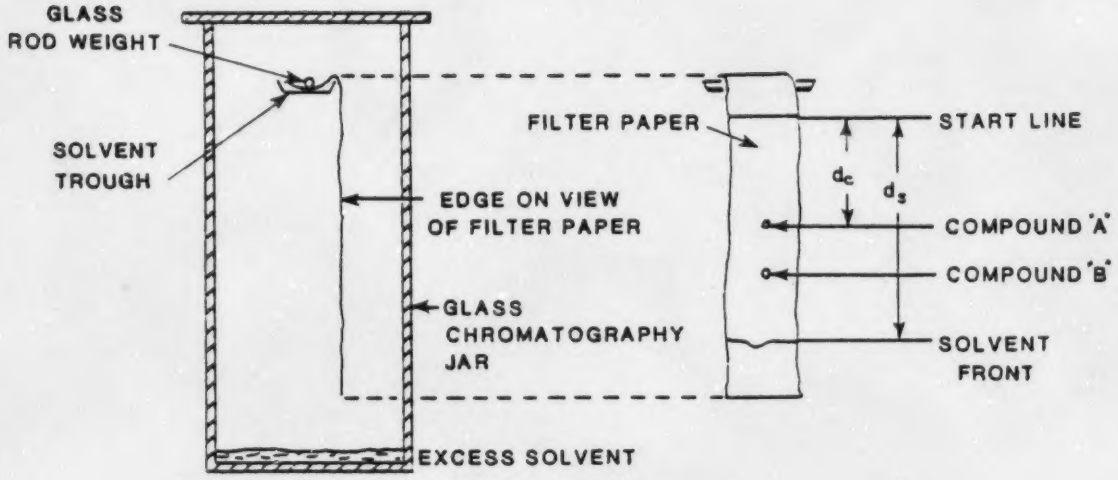

Figure 22-7-6. Descending paper chromatography.

After drying, the paper chromatogram can be read in one of several ways:

- The strip can be placed in contact with a photographic plate to produce an autoradiograph of activity.
- The strip can be cut into small uniform sections and each section assayed.
- The strip can be fed at a slow, constant speed past a detector tied into a ratemeter, which, in turn, is tied into a recorder to furnish a permanent record of the strip's activity.

Thin layer chromatography is procedurally identical to paper chromatography, with the exception of a glass plate coated with a selected adsorbent used instead of the paper strip.

In ion-exchange chromatography, the solution containing the components to be separated is passed through a column of a selected ion-exchange resin, as illustrated in Figure 22-7-7. The result is that all component ions of a given similar charge are held back on the ion-exchange column. Repetitive washing of the column with a suitable solvent will result in separation of the components because of minor differences in ionic character among the components. Each component can then be washed out of the column separately.

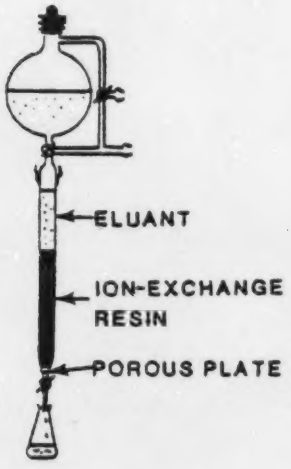

Figure 22-7-7. Ion-exchange chromatography apparatus.

In vapor-phase or gas chromatography, the mobile phase is an inert-carrier gas, such as helium, in which the mixture is carried as a vapor (Figure 22-7-8). As the gas passes over a finely-divided solid phase coated with a hydrocarbon component that has a high-boiling-point and that is essentially nonvolatile, the components are separated according to their relative affinities between the two phases and exit the chromatographic chamber as separated fractions.

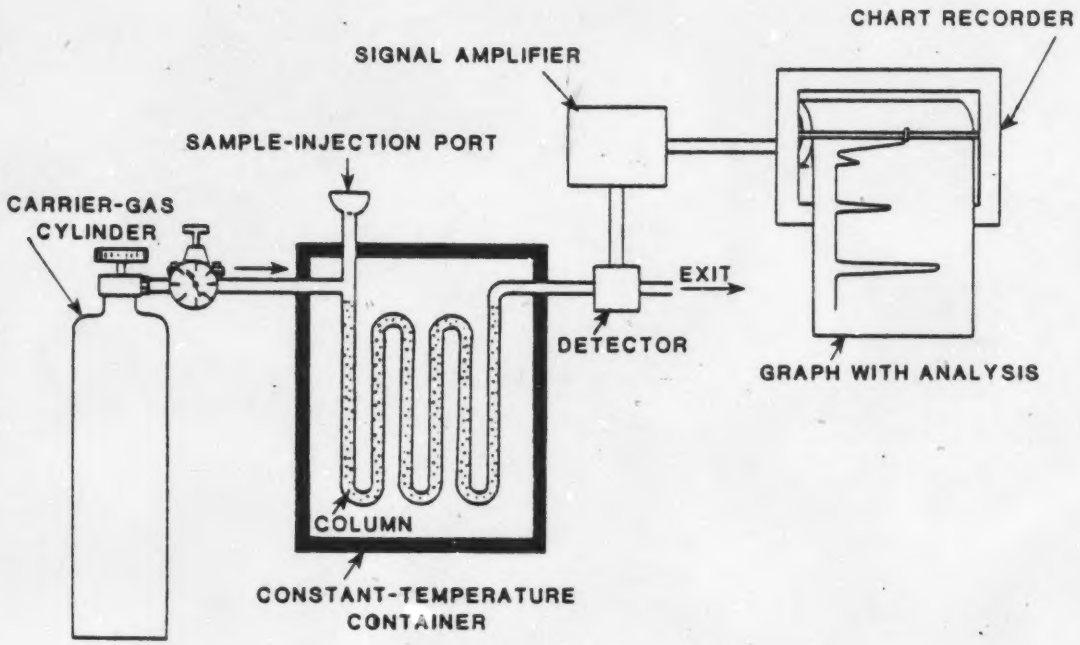

Figure 22-7-8. Vapor-phase or gas chromatography apparatus.

Many compounds can be separated by taking advantage of the difference in vapor pressure among different compounds within a mixture. Distillation is a separatory method that accomplishes this objective. By careful heating of a solution of different substances, each component will be vaporized at a different temperature. These resulting vapor fractions then can be condensed and collected to separate each of the component substances.

Electrochemical Separation

Electrochemical separation is a separatory method based on the migration of ions or colloidal particles in an elec-

tric field. The mixture to be separated is placed in an electrolytic fluid with an electrolytic cell (Figure 22-7-9), and a planchet is used as one of the electrodes within the cell. Depending upon the amount of d.c. voltage applied across the electrodes and upon the polarity of the planchet, any given component of the mixture can be plated out onto the planchet while the remaining components are left in solution.

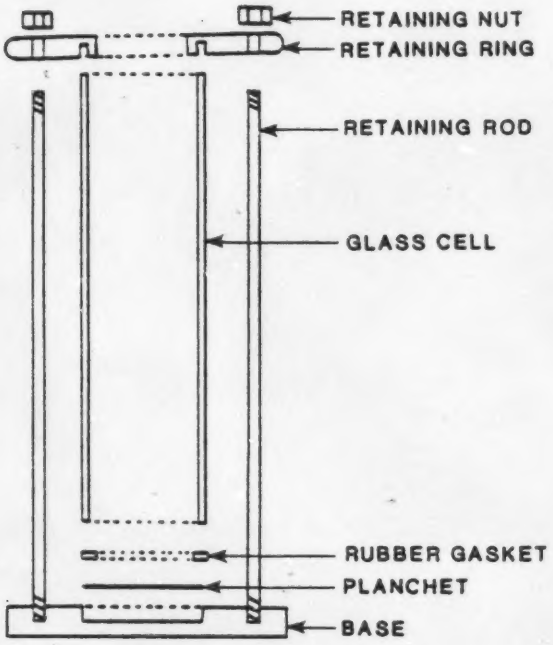

Figure 22-7-9. Electrolytic cell (exploded view).

QUANTITATIVE ANALYSIS OF NUCLIDES

Quantitative analysis usually is performed when the types of nuclides present in the environment already are known. These nuclides may have been identified previously by methods

already discussed or their presence may be suspected because of the activities of the nuclear facility. Power plants, for example, may release fission products such as I, Xe, Cs, Ba, Br, Kr, Rb, and Sr, as well as such activation products as Zn, Co, Cu, Ag, and Fe. Tritium also may be released. A reprocessing facility would release only fission products and tritium; an ore enrichment facility, uranium, thorium, and their daughters; a medical facility, the nuclides it uses. The technician usually knows what to look for.

Sampling procedures are the same as those previously described in Module 22-1. Noncontinuous sampling is used to detect minute traces of radionuclides. Specific techniques depend on whether a technician is sampling for a specific nuclide or for any and all nuclides present.

Once a sample is collected, it is prepared for analysis and its mass and/or its volume carefully measured. It then is identified by qualitative methods and is prepared for counting. The technician must decide upon the nuclides that are to be measured.

When the nuclide(s) is known, its decay scheme is examined to decide upon a counting strategy. Two decay schemes are shown in Figures 22-7-10 and 22-7-11.

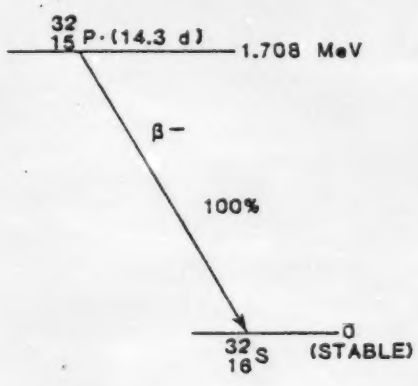

Figure 22-7-10. Decay scheme for $^{32}_{15}P$.

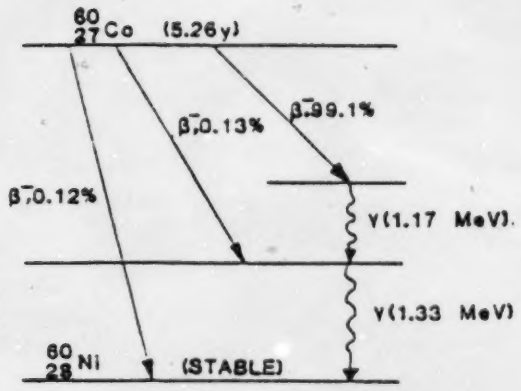

Figure 22-7-11. Decay scheme for $^{60}_{27}Co$.

^{32}P is a pure beta emitter (Figure 22-7-10); therefore, the nuclide must be counted for beta emission. ^{60}Co emits gamma photons of 1.17 MeV and 1.33 MeV (Figure 22-7-11). These gamma rays can be counted and gamma-ray spectroscopy used.

Once a counting strategy is decided upon, the sample is prepared for counting. (Sample preparation was discussed in Module 22-1 and in Course 20.) Exacting preparation of counting samples is as important as the counting operation itself. Several factors affect the choice of a sample form for counting. Tables 22-7-3 and 22-7-4 list procedures for sample preparation of alpha- and beta-emitting nuclides.

TABLE 22-7-3

SAMPLE PREPARATION REQUIRED FOR ALPHA ASSAY

Original Sample Form	Detector	Sample Activity	Sample Preparation	Sample Preparation Time
Gas	Ionization chamber	Low-High	Count directly in gaseous form by introduction into detector in suitable mixture with counting gas.	Moderate
Liquid	Semiconductor	Low-High	Convert to solid thin source and assay.	Moderate-Slow
	Liquid scintillation counter	Low-Medium	Dissolve or suspend in suitable scintillator-solvent mixture and assay.	Quick
Solid	Semiconductor	Low-High	If sample is very thin source, count directly. If not. dissolve sample and redeposit as thin source.	Moderate-Slow
	Liquid scintillation counter	Low-Medium	Dissolve in suitable solvent and heat as liquid, or count directly as a suspension in a gel mixture.	Quick-Moderate
	Nuclear track detectors	Low	Same as semiconductor detector.	Moderate-Slow

For liquid and solid samples, proportional counting is the most popular method for detecting alpha activity and frequently is done at the same time as beta counting.

TABLE 22-7-4

SAMPLE PREPARATION FOR BETA ASSAY

Original Sample Form	Detector	Sample Activity*	Sample Preparation	Sample Preparation Time
Gas	Geiger-Müller proportional counter	Low-Medium Low-High	Sample (1) trapped in suitable solution and converted to solid form, or (2) may be counted directly in gaseous form by introduction into suitable detector chamber with counting gas.	Moderate
	Semiconductor solid scintillation detector	Low-High Medium-High	Trap sample in solution and convert to thin solid source.	Slow
	Liquid scintillation counter	Low-Medium	Must be trapped in suitable solution and treated as liquid sample.	Moderate-Slow
Liquid	Geiger-Müller proportional counter	Low-Medium Low-High	Sample (1) preferably converted to solid form for counting, or (2) may be counted directly with suitable detector if particle energy is sufficiently high.	Moderate-Slow
	Semiconductor solid scintillation detector	Low-High Medium-High	Same as Geiger-Müller counter.	Moderate-Slow
	Liquid scintillation counter	Low-Medium	May be dissolved or suspended in a suitable scintillation solvent mixture and assayed directly without pretreatment.	Quick
Solid	Geiger-Müller proportional counter	Low-Medium Low-High	May be counted directly unless low energy of particles requires pretreatment of sample.	Quick
	Semiconductor solid scintillation counter	Low-High Medium-High	Same as Geiger-Müller counter.	Quick
	Liquid scintillation counter	Low-Medium	(1) Should be dissolved in suitable solvent and treated as liquid sample, or (2) may be counted directly as suspension in a suitable gel mixture, or (3) in the case of ^{3}H and ^{14}C, samples can be burned to H_2O and CO_2, which can be dissolved or trapped as in (1).	Moderate-Slow

* Low = < 1pCi
Medium = 1 uCi
High = > 1 mCi

Biological samples also must be converted to suitable counting forms. The specific treatment given each sample depends upon its original form, the radiation energy, and the type of detector used.

Ashing is the most practical method of preparing a biological sample in which the radionuclide is metallic or mineral. Several combustion methods are used, and unique methods specifically designed for combustion of samples for liquid scintillation counting exist.

When the material is prepared in a suitable counting form, the sample is counted.

Samples to be counted in a gaseous form often are introduced directly into the detector. Gas counting reduces self-absorption and is used widely in the analysis of low-energy β^- emitters such as ^{14}C, ^{3}H, and ^{35}S.

Liquid sample counting is restricted mainly to gamma-ray emitters and high-energy β^- emitters. NaI or GeLi semiconductor detectors usually are employed for gamma-ray counting of liquid samples.

Samples in solid form frequently are preferred to other types since they can be stored and handled more easily. Methods for the preparation of solid samples vary and include direct evaporation, filtration, settling, electroplating, and vacuum evaporation.

After preparation, the sample is counted by the appropriate method. Counts then are converted into count rate; statistical adjustments are made as necessary; and the activity of the sample is calculated from Equation 22-7-1.

$$A = \frac{C}{(BR)(\Sigma)} \qquad \text{Equation 22-7-1 (Equation = Eq.)}$$

where

A = activity of sample.

C = count rate.

BR = branching ratio.

Σ = counting efficiency.

The activity then is converted to units of activity, and the concentration is calculated by division of the mass or volume of the sample into curies.

EXAMPLE

Given: A 100-ml sample of water is counted for its ^{60}Co concentration. The NaI counter shows the 1.33-MeV count rate to be 100 cps, with a counting efficiency of 0.05.

Find: The concentration (in pCi per ml) of ^{60}Co.

Solution: From Equation 22-7-1:

$$A = \frac{100 \text{ cps}}{(0.991)(0.05)}$$

From Fig. 22-7-11 the BR = 0.991.

$$A = 2018 \text{ dps}$$

$$\text{Concentration} = \frac{2018 \text{ dps}}{(100 \text{ ml})(3.7 \times 10^{-2} \text{ dps/pCi})}$$

$$= 5.45 \times 10^{2} \text{ pCi}$$

Some general procedural examples for determining the activity of specific nuclides in the environment are summarized in Tables 22-7-5 through 22-7-7.

TABLE 22-7-5

^{60}Co IN SEDIMENT OR SOIL SAMPLES

1.	Examine decay properties: ^{60}Co has two gamma rays with very good branching ratios. If a NaI and/or GeLi detector with multichannel analyzer is available, plan to use, and count the gammas.
2.	Collect soil sample.
3.	Sieve, dry, and wash.
4.	Count on detector.
5.	Integrate the 1.33- or 1.17-MeV gamma ray.
6.	Calculate the counts per time.
7.	Convert to activity and, finally, to concentration expressed in curies per gram.
8.	Compare with either a control sample or a predicted maximum concentration.

TABLE 22-7-6

^{90}Sr IN VEGETATION

1.	Examine decay scheme. Since a beta count will be necessary, all other beta emitters must be eliminated by chemistry.
2.	Collect leafy vegetation, dry, and weigh.
3.	Dissolve in hot acid (HCl and HNO_3).
4.	Add 40 mg of SR in solution as an Sr carrier. All of the ^{90}Sr will travel chemically with the inactive Sr that has been added.
5.	Add H_2SO_4 to precipitate $SrSO_4$.
6.	Centrifuge acids. Filter and dry.
7.	Weigh the precipitate, and calculate a chemical yield. (Yield = mg Sr in precipitate/mg Sr added in Step 4.)

8.	Count.
9.	Calculate the count rate; correct for the chemical yield; and convert to activity and concentration of ^{90}Sr in dry vegetation.
10.	Compare with expected norm or control sample.

TABLE 22-7-7

TRITIUM IN WATER SAMPLE

1.	Examine decay scheme. Since only weak betas are present, a liquid scintillation count must be done. This process requires the elimination of all other beta emitters. When the water is distilled, all other nonvolatile radionuclides will be left behind.
2.	Collect sample, and filter out matter.
3.	Distill water using still.
4.	Take a known volume of distilled water, and make a liquid scintillation counting cocktail.
5.	Count.
6.	Convert count rate to disintegration rate, using efficiency quench curve.
7.	Convert disintegration rate to activity per volume of water in cocktail in units of curies/liter.
8.	Compare with control sample.

EXERCISES

1. Describe in detail how the concentration of ^{137}Cs in vegetation is determined.
2. How much activity per gram exists in 1.8 g of sediment beta counted on a 20%-efficiency counter at a count rate of 168 cpm net?
3. Explain why particulate matter must be filtered out of liquid samples before any counting is done.
4. Explain why water must be distilled, or otherwise chemically purified, before any determination for tritium.

MATERIALS

Soil samples that contain a very small amount of ^{137}Cs.
Liquid sample that contains a small quantity of ^{90}Sr.
Appropriate counters.
Appropriate chemicals.

PROCEDURES

DETERMINATION OF ^{137}Cs IN SOIL

NOTE: Record data for this procedure in Data Table 22-7-1.

1. Examine the decay scheme of ^{137}Cs (along with ^{137m}Ba) to determine the best way to count the sample.
2. Take the dirt sample given and dry it.
3. Weigh a portion of the sample suitable for counting.

4. Prepare the sample for counting.
5. Count in a geometry with a known counting efficiency.
6. Calculate the count rate for ^{137}Cs.
7. Determine the activity per gram of sample, and compare the answer with the concentration given by the instructor.

DETERMINATION OF ^{90}Sr IN LIQUID

NOTE: Record data for this experiment in Data Table 22-7-2.

1. Look up the decay properties of ^{90}Sr, and decide how to count.
2. Take the liquid sample given by instructor; filter it if necessary. Measure the volume of liquid.
3. Add about 40 mg of Sr in solution; mix thoroughly.
4. Add a small amount (10 ml of 5M) of H_2SO_4; mix thoroughly.
5. After letting the $SrSO_4$ coagulate for several minutes, centrifuge the sample. Add more H_2SO_4 to the supernate to ensure that all the Sr has been precipitated.
6. Filter the precipitate, and weigh it.
7. Determine the chemical yield.
8. Prepare the precipitate on the filter for counting.
9. Count the sample.
10. Determine the count rate of the sample.
11. Calculate the activity, using the counting efficiency, chemical yield and branching ratio.
12. Calculate the activity per liter of liquid used.

DATA TABLES

DATA TABLE 22-7-1
DETERMINATION OF ^{137}Cs IN SOIL

Decay properties (branching ratio and mode of decay)
_____
Mass of sample _____
Preparation and/or chemical separation data _____
Count rate _____
Counting efficiency _____
Decay rate _____
Activity of sample _____
Activity of sample per gram _____

DATA TABLE 22-7-2
DETERMINATION OF ^{90}Sr IN LIQUID

Decay properties (branching ratio and mode of decay)
_____
Volume of sample _____.
Preparation and/or chemical separation data _____
Count rate _____
Counting efficiency _____
Decay rate _____
Activity of sample _____
Activity of sample per liter _____

REFERENCE MATERIALS

Chase, Grafton D., and Rabinowitz, Joseph L. Principles of Radioisotope Methodology. 3rd ed. Minneapolis: Burgess Publishing, 1967.

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Wang, C.H.; Willis, D.; and Loveland, W.D. Radiotracer Methodology in the Biological, Environmental, and Physical Science. Englewood Cliffs, NJ: Prentice Hall, 1975.

Waser, Jürg; Trueblood, Kenneth N.; and Knobler, Charles M. Chem One. New York: McGraw-Hill Book Co., 1976.

Weast, Robert C., ed. CRC Handbook of Chemistry and Physics. 55th ed. Cleveland, OH: CRC Press, Inc., 1974. (This work is updated yearly.)

PART I: MULTIPLE CHOICE.

Choose the best response(s) to the following statements by circling the letters:

1. Qualitative analysis is used to –
   a. Determine the identity of a radionuclide.
   b. Determine the concentration of activity of a radionuclide.
   c. Chemically separate radioactive isotopes.
   d. Both identify and determine the activity of a radionuclide.
2. Radionuclides may be identified by –
   a. Measuring their half-life.
   b. Measuring the energy of the radiation.
   c. Chemical analysis.
   d. Identifying the types of radiation emitted.
3. A pure beta emitter has an approximate energy of 1.2 MeV and a half-life of from 1-6 hours. The beta emitter is (use Table 22-7-1) –
   a. ^{91}Y.
   b. ^{99}Mo.
   c. ^{41}A.
   d. ^{31}Si.
4. Which of the following nuclides probably would be present after an environmental release from a nuclear power plant?
   a. ^{60}Co.
   b. ^{224}Ac.
   c. ^{220}Fr.
   d. ^{210}Pb.

PART II: STATEMENT COMPLETION.

5. An accidental release of ^{99}Mo into a hospital sewer system is suspected. Describe in detail the strategy (a) to determine if any ^{99}Mo is present in a sample of sewer water (i.e., qualitative analysis) and (b) to determine how much ^{99}Mo is present (i.e., quantitative analysis).
   This question is concerned more with the difference in the strategies between (a) and (b) than with the details in the determination of (a) and (b) individually.

6. The concentration of tritium in water can be determined as follows:

7. The concentration of ^{90}Sr in vegetation can be determined as follows:

8. The concentration of ^{60}Co in sediment or soil can be determined as follows:

9. The concentration of ^{140}Ba can be determined as follows:

PART III: CALCULATIONS.

Given: A 55.3-g sample of sieved sediment counted by a detector of 3.5% efficiency. Net counts in the 1.33-MeV gamma-ray peaks of 347. Length of count: 128 minutes.

Find: The picocuries of ^{60}Co per gram of sediment.

Solution:

NUCLEAR TECHNOLOGY

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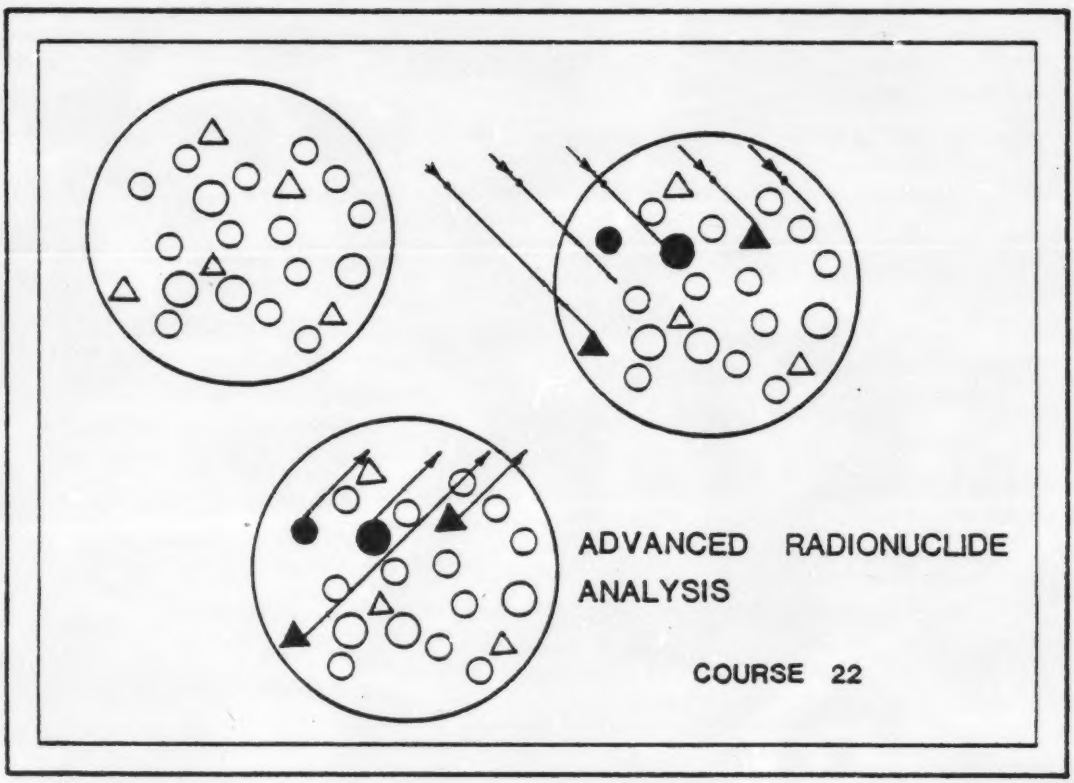
COURSE 22 - ADVANCED RADIONUCLIDE ANALYSIS



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for

COURSE 22, ADVANCED RADIONUCLIDE ANALYSIS (SEVEN MODULES)

NOTE: Each module in this Course is numbered separately, and the page numbers begin with page 1 in each module. Therefore, this Index lists for each entry the module number (underlined), followed by a comma and the page number. Figures and tables are listed by their numbers, followed by the module and page number in parentheses. A Master Index is available in a separate volume. This general index merges all index entries from the 35 Nuclear Courses. It is a valuable research tool, with which the student or researcher can locate all the references to any subject within the 35 Courses.

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